*University of Alberta*
*Printing Department*

Digitized by the Internet Archive
in 2019 with funding from
University of Alberta Libraries

https://archive.org/details/Sorensen1965

Thesis
1965
#78

THE UNIVERSITY OF ALBERTA

# THE SULPHUR STATUS OF SOILS IN NORTH-CENTRAL ALBERTA

by

ALLAN KINGSLEY SORENSEN, B. Sc.

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES

IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE

OF MASTER OF SCIENCE

DEPARTMENT OF SOIL SCIENCE

EDMONTON, ALBERTA

APRIL, 1965

UNIVERSITY OF ALBERTA

FACULTY OF GRADUATE STUDIES

The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for acceptance, a thesis entitled "The Sulphur Status of Soils in North-Central Alberta" submitted by Allan Kingsley Sorensen, B. Sc. in partial fulfilment of the requirements for the degree of Master of Science.

ABSTRACT

Sodium sulphate was applied in 1963 and 1964 at the rate of 20 lb. elemental sulphur/acre to 133 forage stands containing at least 25 per cent legume on predominately Grey Wooded and Dark Grey soils in north-central Alberta. The legume responded to the applied sulphur on 34 plots. Moisture conditions were subnormal for the 2 years and it was therefore suggested that the above is an underestimate of the extent of the sulphur-deficient soils in north-central Alberta.

Sulphur deficiency was not confined to any geographic area nor to any soil series. However, certain soils series exhibited sulphur deficiency more frequently than did others.

The ethanol-soluble sulphate of the legumes grown on sulphur-deficient soils was lower than in legumes grown on sulphur-sufficient soils. Taking 0.17 per cent ethanol-soluble sulphate as the critical level for alfalfa, deficient and non-deficient soils could be separated with 84 per cent reliability.

Sulphur application increased the nitrogen content of the legumes grown on the deficient soils by an average of 0.52 per cent. No difference was found in the nitrogen content of the legumes grown on non-deficient soils as a consequence of the sulphur application.

Four sulphur-deficient and 4 non-deficient soils were investigated at 4 depths for total sulphur, carbon-bonded sulphur, HI-reducible sulphur, sulphate extractable by a 500 ppm P solution, adsorbed sulphate and easily-soluble sulphate. The non-deficient soils had significantly greater amounts of HI-reducible sulphur, sulphate extractable by a 500 ppm P solution, and

easily soluble sulphate than did the deficient soils. The easily soluble sulphate content of the soil gave the best separation of the sulphur-deficient and sulphur non-deficient soils. The total sulphur content, carbon-bonded sulphur, and adsorbed sulphate did not differ significantly between deficient and non-deficient profiles. The sulphur content of all fractions studied was low compared to chernozems.

ACKNOWLEDGEMENTS

Sincere appreciation is extended to Dr. J. A. Robertson for his thoughtful guidance throughout the course of this study and his constructive criticisms in the preparation of the manuscript.

Thanks are also extended to Dr. L. E. Lowe of the Research Council of Alberta for his suggestions in some of the chemical analyses and for serving on the committee, and to Dr. J. A. Toogood for his helpful suggestions.

Acknowledgements are extended to the following: Mr. A. A. Kjearsgaard of the Canada Department of Agriculture for assistance in the classification of the soils; Mr. D. H. Laverty of the Soil and Feed Testing Laboratory for assistance with some of the chemical analyses; Dr. W. F. Allen of the Department of Chemistry and Dr. G. R. Webster for serving on the committee; Peter Lupul of the Research Council of Alberta for assistance with some of the chemical analyses; and members of the Department of Soil Science for help from time to time. The financial assistance of Federated Co-operative Ltd., Harrisons and Crossfield (Canada) Ltd., Northwest Nitro-Chemical Ltd., and Sherritt-Gordon Mines Ltd. has been appreciated.

Gratitude is expressed to Mrs. D. H. Laverty for her efficient typing of the manuscript and to the author's fiancee, Miss Doris York, for help with the typing and for her patience and encouragement during the course of this study.

TABLE OF CONTENTS


| | Page |
|---|---|
| INTRODUCTION | 1 |
| REVIEW OF LITERATURE | 2 |
| Role of Sulphur in Plant Nutrition | 2 |
| Sulphur Balance in the Soil | 3 |
| Sulphur Additions | 3 |
| Sulphur Removal | 4 |
| Sulphur Deficiency in Soils | 5 |
| Areas of Sulphur Deficiency | 5 |
| Crops Affected by Sulphur Deficiency | 6 |
| Sulphur Containing Fertilizers | 6 |
| Forms of Sulphur in the Soil | 7 |
| Total Sulphur | 7 |
| Organic Sulphur | 7 |
| Carbon-Bonded Sulphur | 8 |
| Organic Sulphate | 9 |
| Inorganic Sulphur | 9 |
| Adsorbed Sulphate | 10 |
| Methods of Determining Sulphur | 11 |
| Methods of Estimating the Sulphur Status of the Soil | 11 |
| Total Sulphur | 11 |
| Extraction Methods | 12 |
| HI-Reducible Sulphur | 13 |
| Plant Analyses Methods | 13 |
| Summary of the Literature Review | 14 |
| MATERIAL AND METHODS | 15 |
| Field Plots | 15 |
| Laboratory Analyses | 24 |
| Plant Samples | 24 |
| Soil Samples | 24 |
| RESULTS AND DISCUSSION | 30 |
| Field Plots | 30 |
| Responses of the Legume Crops | 30 |
| Moisture Conditions | 38 |
| Available Nutrients | 39 |
| Laboratory Analyses | 41 |
| Plant Samples | 41 |
| Soil Samples | 46 |
| SUMMARY AND CONCLUSIONS | 56 |
| BIBLIOGRAPHY | 58 |
| APPENDIX | 66 |

LIST OF TABLES


Page

1. Classification and Location of Soils . . . . . 18

2. Soils Used in Comprehensive Soil Study . . . . . 27

3. Yields, Responses, and Available Nutrients of the 1963 Sites . 31

4. Yields, Responses, and Available Nutrients of the 1964 Sites . 34

5. Ethanol-soluble Sulphate of the Legumes . . . . . 42

6. Nitrogen Content of the Legumes . . . . . 45

7. Texture, Total Carbon, Nitrogen Content, and pH of Selected Profiles . . . . . 47

8. Sulphur Fractions of the Selected Profiles . . . . . 49

9. Summary Table of the Sulphur Fractions . . . . . 50

## LIST OF FIGURES

Page

1. Map showing area studied, plot sites, and the response of the legumes to sulphur fertilization . . . . . 16

2. HI-Reducible sulphur, extractable sulphate, and easily-soluble sulphate of sulphur-deficient and non-deficient soils . . . . . 52

## LIST OF APPENDICES


Page

1. Soil Moisture, 1/3 Atmosphere Percentage, and Rainfall Data for 1963 . . . . . 66

2. Soil Moisture and Rainfall for 1964 . . . . . 69

# INTRODUCTION

The essentiality of sulphur in plant nutrition has long been known; however, sulphur has been called the "neglected nutrient" in plant nutrition.

All Grey Wooded soils in Alberta were thought to be sulphur deficient after Newton (1936) so conclusively demonstrated this deficiency at Breton in the early 1930's. After that time limited evidence suggested that not all Grey Wooded soils were deficient in sulphur.

Walker (1959) found only 44 per cent of the soils he investigated were sulphur deficient in an area generally south and west of Edmonton. He also found that the sulphur deficient soils were not confined to any geographic area. Responses to sulphur in Alberta have been demonstrated only with legume crops, although there are some unpublished reports of cereals responding to sulphur fertilization.

There appeared to be no reliable chemical methods of predicting the sulphur status of the soil. It has been found by several workers that legumes contain less easily-soluble sulphur when grown on sulphur deficient soil, thus providing a method of assessing the sulphur status of the soil (Walker, 1959; Dijshoorn et al., 1960; Fox and Olson et al., 1964).

The purpose of this investigation was to study the sulphur status of some soils in the Grey Wooded and Dark Grey soil zones of north-central Alberta under the following headings:

1. The extent of sulphur deficiency in these soils and the geographic distribution.

2. The assessment of the sulphur status of the soil by plant analyses.

3. A study of various sulphur fractions of the soil to determine if differences in these fractions existed between deficient and non-deficient soils and if these differences would be useful for prediction purposes.

## REVIEW OF LITERATURE

Sulphur is an essential plant nutrient. It is a constituent of methionine, cystine, and cysteine, three essential amino acids. Two vitamins, thiamine and biotin, contain sulphur. Hence a close connection exists between sulphur and nitrogen (Thomas et al., 1950b). Although sulphur is not a constituent of chlorophyll, sulphur deficient plants become chlorotic. Ergle (1953) found a 40 per cent reduction in the chlorophyll content of sulphur deficient plants. Thomas et al. (1950b) suggested that sulphur is also involved directly in chlorophyll metabolism through the protoplasm of the chloroplasts.

Sulphur is required in large amounts, approaching and often exceeding that of phosphorus (Jordan and Ensminger, 1958). Thomas (1950a) found that the total sulphur content of alfalfa leaves differs by a factor of 12, the average being 0.69 per cent. A three ton/acre crop of alfalfa contains on the average 20 lb./acre of sulphur in the plant tops. He also found cereals grown in areas far removed from industrial centers to contain an average 0.39 per cent total sulphur in the leaves.

The most obvious sign of a sulphur deficiency is the chlorotic, stunted growth (Jordan and Ensminger, 1958). The stems of sulphur deficient plants are inclined to be woody (Ergle and Eaton, 1951). Leaf area is reduced. Chlorosis may involve the whole plant or it may be severe only on the younger leaves. Sulphur deficient plants resemble those which are nitrogen deficient except that they do not develop the characteristic leaf patterns as is usual with nitrogen deficiency. The weight and number of nodules on legumes are reduced (Anderson and Spencer, 1950). They considered this a reflection of reduced growth and the consequent lower demand for nitrogen.

## Sulphur Balance in the Soil

Fertilizers, manures, irrigation and ground waters, gaseous sulphur, insecticides, and fungicides may contribute significant amounts of sulphur to the soil over restricted areas of the earth's surface (Bentley et al., 1955; Freney et al., 1962). Rainfall contributes a variable amount over the entire earth. In many areas of the world sulphur in the rain water is sufficient to meet the demands of the crop (Alway et al., 1937; Lutz, 1956; Miller, 1958). Bentley et al. (1955) reported 0.8 lb. of sulphur/acre at Breton and 4.9 lb./acre at Edmonton were brought down in the precipitation during the growing season. Alway found a range from 5 to 100+ lb./acre/year in the rain water of Minnesota. In Virginia ranges of 12.9 to 33.5 lb. of sulphur/acre/year are recorded (Lutz, 1957). Miller (1957) reported 2.8 to 4 lb. of sulphur/acre/year in rural areas and an average of 12 lb./acre/year near industrial plants in New Zealand.

Junge and Wirby (1958) reported wide variations in the sulphur content of the precipitation and they considered sufficient information was available to report averages on a world wide basis. They gave the average for the United States as 2.15 mg./l., for Scandinavian countries as 2.9 mg./l., and for the earth's land surface as 2.2 mg./l. With a rainfall of 18 in./year, this would represent 6 lb. of sulphur/acre/year. This is a substantial amount compared to crop needs. Martin (1958) stated that 5 to 6 lb. of sulphur are required per ton of alfalfa harvested.

In all reports, areas near industrial plants have the highest concentrations of sulphur in the precipitation. The pattern of distribution depends on the prevailing wind (Jordan and Ensminger, 1958). They showed that the operation of one steam plant in Alabama added 89,000 tons of sulphur per year to the atmosphere. The sulphur content in the rain-

water increased from 5 to 10 lb./acre/year because of the steam plant. According to Junge (1958) 30 per cent of the sulphur in the air originates from human activities. He estimated that industrial plants in the world added $38.677 \times 10^6$ tons of sulphur to the atmosphere in 1943.

Irrigation waters can be an important source of sulphur. Thorne and Peterson (1954) summarized the chemical composition of water from 19 rivers used for irrigation in the western United States and found the sulphur content ranged from 3 to 1,849 lb./acre foot. The majority of the waters contained more than 40 lb. of sulphur per acre foot. Jensen (1951) showed well water used for irrigation in Idaho ranged from 25 to 700 lb. of sulphur per acre foot.

The atmosphere contains $SO_2$ and $SO_3$ which may be adsorbed by the soil (Alway et al., 1937). They found that a surface coated with lead peroxide accumulated the equivalent of 400 lb./acre/year in Minnesota. They concluded that sulphur adsorbed by the soil made an important contribution to plants, but that the amount would likely be less than in the rain water. It has been found by Jordan and Ensminger (1958) that soils in Alabama and Virginia adsorbed 5.5 lb./acre and 11 lb./acre, respectively.

There are continuous losses of sulphur which offset the additions. Some early lysimeter studies showed very large losses by leaching. In Illinois, measured losses ranged from 35 to 55 lb./acre/year (Illinois Agr. Exp. Sta., 1937). McKill and Williams (1960) used $S^{35}$ to study leaching. They found that 77 per cent of the sulphur applied as gypsum was recovered in the percolate at a depth of 25 inches. Stauffer and Rust (1954) showed that the loss of sulphur depended greatly on the amount of percolate. They found a range in losses from 5 to 80 lb./acre/year when the percolate varied from 1 to 20 inches. The losses are also dependent on other

characteristics of the soil such as texture and compaction (Stauffer and Rust, 1954). Losses of sulphur by leaching were high in soils well supplied with native sulphur while losses were much more moderate when native sulphur was low.

Sulphur may be lost from the soil as a result of microbial transformation (Jordan and Ensminger, 1958). Under aerobic conditions sulphur is oxidized to sulphate which is easily leached. Under anaerobic conditions reduced forms such as sulphides predominate, and some losses may result through volatilization as hydrogen sulphide.

Cultivated soils continually suffer some loss of sulphur as a result of erosion. Lipman and Conybeare (1936) estimated that as an average for the United States about 6 lb. of sulphur per acre/year are lost in this manner. Any plant material removed also represents a loss of sulphur.

## Sulphur Deficiency in Soils

Sulphur deficiency in soils is widespread. Plant responses to applied sulphur have been reported in South Africa (Goldschmit, 1951), Nigeria (Greenwood, 1955), Australia (Anderson, 1952; Rossiter, 1952; McLachlan, 1955; Spencer, 1960), Canada (Bentley *et al.*, 1955; Walker and Bentley, 1961; Alberta Advisory Fertilizer Committee, 1964), Burma (Aiyar, 1945), Brazil (McClung, 1959), New Zealand (Anderson, 1952), Japan (Araki, 1954), West Indies (Dutt, 1962), Sweden (Gunnarsson, 1959), and the United States (Jordan and Ensminger, 1958; Martin, 1959).

Sulphur deficiency was first reported on a Grey Wooded soil in Alberta by Newton (1936). Walker and Bentley (1961) reported responses at 72 of 157 test locations in an area west of a line between Edmonton and Calgary.

In general legumes have shown the greatest sulphur response because they are the heaviest users and because they are so widely grown. More moderate responses have been shown by cereals, onions, rice, jute, and other crops. In Alberta responses from sulphur applications have been most common on legume crops.

Sulphur deficiency bears no relationship to geographic location (Walker and Bentley, 1961; McLachlan, 1955) although crops grown on certain soil types are known to respond to sulphur (Newton et al., 1936; Carins and Richer, 1960).

McClung and de Freitas (1959) found that crops grown on Brazilian soils did not respond to sulphur unless phosphorus was also added. Stephens and Donald (1958) reported that sulphur deficiencies are often related to phosphorus deficiencies. In some cases sulphur must be applied before other nutrient deficiencies are manifested, while in other instances certain nutrients must be applied before sulphur deficiencies are observed. Residual effects of sulphur are common (Martin, 1963).

Sulphur has been found to be equally effective when applied as $CaSO_4$, $Na_2SO_4$, or as 8 mesh elemental sulphur. However, the oxidation of elemental sulphur required about one year (Fox and Atesalp et al., 1964). Utilization of elemental sulphur was therefore slower, although Fox and Atesalp et al. (1964) found finely divided elemental sulphur to be utilized as fast as gypsum in a greenhouse study. The incidental addition of sulphur in fertilizers has been, and will continue to be, an important source of sulphur for crop production (Jordan and Ensminger, 1958). Superphosphate contains 12.2 per cent sulphur and ammonium sulphate contains 23.9 per cent sulphur (Mehring et al., 1950). Other fertilizers contain lesser amounts.

## Forms of Sulphur in the Soil

The total sulphur content in the soil is extremely variable. Lawton (1955) reported sulphur in mineral soils of the humid areas range from 0.01 to 0.15 per cent. Wyatt and Doughty (1928) reported a range of 0.03 to 0.07 per cent for Alberta soils and Bentley et al. (1955) found a similar range. Lowe (1965) found a range in the total sulphur content from 0.006 to 0.10 per cent for the upper horizons of some Alberta soils. The high figures represented the L-H horizon and the low figures the Bt horizon of a Grey Wooded soil.

Soil sulphur exists in both organic and inorganic forms. The organic form is derived from the protein material of plant residues (Evans and Rost, 1945). Other organic compounds, including taurine and mustard oils, may introduce variable amounts of sulphur to the soil (Waksman, 1947). The organic fraction is present in amounts from 50 to 90 per cent of the total. Organic sulphur is generally estimated as the difference between total sulphur and inorganic sulphate (Freney et al., 1962). Only a few workers have attempted to measure the organic fraction directly (Madanov, 1946).

There appears to be a definite relationship between carbon, nitrogen, and non-sulphate sulphur in the soil (Donald and Williams, 1954; Walker and Adams, 1958; Freney, 1961). According to Walker and Adams, the average total C:N:organic P:S ratio for 20 soils to a depth of 21 inches was 120:10:2.7:1.3. Using nitrogen as a base with the value 10 total C ranged from 70 to 170, sulphur ranged from 0.7 to 2.1, and organic phosphorus ranged from 1.2 to 5.3; however, most of the values were close to the mean. They concluded since phosphorus is the only element of the four which must be supplied by the parent material, the major factor governing the

accumulation of organic matter, and hence organic sulphur, is the phosphorus content of the parent material.

Very little is known about the sulphur compounds which constitute soil organic matter. Trithiobenzaldehyde and cystine are the only organic sulphur compounds to have been extracted from the soil, although cysteine, cystine, and methionine have been detected after acid hydrolyses (Paul, 1961; Whitehead, 1964). Whitehead (1964) calculated that 11 to 16 per cent of the total sulphur in the soil is in the amino acid form. He stated that, since a much higher percentage of the total sulphur is in protein form when added by plant, animal and microbial remains, and that since amino acids and proteins are easily decomposed when added to the soil alone, some stabilization of the sulphur must occur during the formation of humus. In aerobic conditions cysteine is decomposed mainly to sulphate while methionine decomposition results in a substantial release of mercaptans (Starkey et al., 1953). There is evidence that the complete decomposition of methionine to sulphate takes place over a longer period of time than cystine (Hesse, 1957). In contrast Frederick et al. (1957) found no sulphate formation from methionine after six weeks.

The group whose chemical reactions are the most important in the incorporation of sulphur into humus appears to be the sulphhydryl group (Whitehead, 1964). Since most of the nitrogen added to the soil under natural conditions occurs in amino acid form, sulphur and nitrogen may be stabilized into humus in similar ways (Whitehead, 1964).

A fraction designated as "carbon bonded sulphur" has been investigated by Lowe and DeLong (1963). This fraction includes all sulphur bonded to carbon except the alkyl sulphones. It does not include organic sulphate. Elemental sulphur and some mineral sulphides are included

although of the soils they investigated no measurable amounts of the latter materials were present. They suggest that much of the carbon-bonded sulphur may be associated with the humic acids. No information is available at the present time on the availability of this fraction to plants.

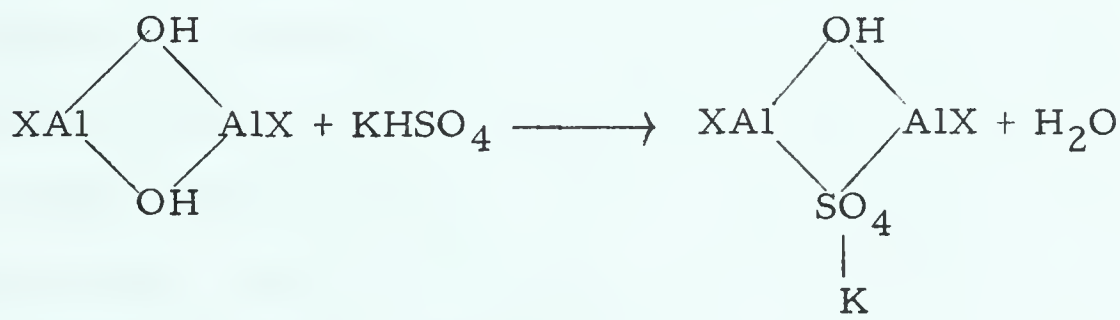
Another form of sulphur in the soil is organic sulphate (Freney, 1961; Delong and Lowe, 1962). There is no sharp distinction between organic and inorganic sulphate. Freney (1961) has postulated that a significant portion of total sulphur exists as sulphate that is not simply adsorbed, but forms an integral part of soil organic matter.

Inorganic sulphur is present in soils in amounts from 10 to 50 per cent of the total sulphur (Lawton, 1955). Most unweathered igneous rock contains 0.05 to 0.3 per cent sulphur, mainly as sulphides of iron, nickel, and copper. In strongly reducing conditions sulphides accumulate in soils (Hart, 1962). Generally most of the inorganic sulphur is in forms of sulphate. In 24 agricultural soils Freney (1961) found only 1 per cent of the sulphur in a form of lower oxidation state than sulphate.

Of the sulphates, those of barium and strontium are extremely insoluble. Calcium sulphate has a low but significant solubility and is steadily removed by leaching if the net movement of water is downward. The sulphates of magnesium, potassium, and sodium are easily soluble. Sulphate resistant to leaching has been found associated with calcium carbonate and may constitute a major proportion of the total sulphur in some calcareous soils (Williams and Steinbergs, 1962). Lowe (1965) has found the amount of sulphate associated with calcium carbonate to be low in the Grey Wooded soils of Alberta.

Most workers have found that little retention of sulphate occurs in the top few inches of the soil (Ensminger, 1954; Hesse, 1958; Freney, 1961),

but considerable sulphate is retained in the lower depths (Ensminger, 1958; Jordan and Bardsley, 1958; Chao *et al.*, 1962a). Retained or adsorbed sulphate is not removed with water extractions. The main soil constituents involved in sulphate retention are hydrated aluminum and iron oxides, especially at low pH values and kaolinitic clays (Chao *et al.*, 1962a). The same workers found that treatments involving the removal of iron and aluminum hydroxides resulted in a marked reduction in sulphate retention. Several mechanisms by which sulphate may be held have been suggested. Jackson (1963) suggests that in both hydrated aluminum oxides and the alumina layer of kaolinite the following type of reaction can occur:




It has been suggested that sulphate may be held by simple anion exchange (Schell and Jordan, 1958). However, Chao *et al.* (1962c) indicated that soils do not have a definite anion exchange capacity, and therefore mechanisms other than simple anion exchange must be involved. Early work of Mattson (1931) showed that anion adsorption increased with a decrease in pH. Mattson (1931) found the effectiveness of anions replacing hydroxyl groups was in the order: phosphate>sulphate>chloride. Williams and Steinbergs (1962) found no relationship between the amount of clay and anion adsorption. Ensminger (1954) demonstrated that superphosphate reduced the amount of sulphate retained in the surface 6 inches.

Under anaerobic conditions sulphates may be reduced to hydrogen sulphide (Waksman and Starkey, 1947). This reduction is brought about mainly by the bacterial genus *Desulphovibrio*. The oxidation of sulphur to

sulphate can occur in purely chemical condtions, but microbial oxidation is more important. The genus Thiobacillus is the most prominent group of bacteria in this respect (Starkey, 1950).

## Methods of Determining Sulphur

Sulphate can be determined gravimetrically, volumetrically, turbidimetrically, electrometrically, or colorimetrically (Johnson and Nishita, 1952; Cassidy, 1956; Asghar et al., 1957; Bertolacini and Barney, 1957). In spite of the variety of methods available, no one method has been widely accepted by soil scientists. The gravimetric method is time consuming and magnitude of the errors often equals the difference between samples (Chesnin and Yien, 1950). Turbidimetric methods lack the necessary accuracy for sulphur determinations at the low concentrations found in the soil. The colorimetric method of Johnson and Nishita (1952) is a micro method and has high precision. Consequently, many workers have adopted the method (Williams and Steinbergs, 1959; Freney, 1961; Lowe and DeLong, 1961).

## Estimating the Sulphur Status of the Soil

Diagnostic methods for sulphur status have followed the general approaches used for other elements such as nitrogen and phosphorus, which include extraction by various solvents, incubation, microbial growth, plant growth, and plant composition (Freney et al., 1961).

Total sulphur is a poor index of available sulphur (Bardsley and Lancaster, 1960; Spencer and Freney, 1960; Walker and Bentley, 1961). Only moderate success has been achieved with procedures which displace sulphate or soluble sulphur from the soils. Spencer and Freney (1960) compared eight methods of estimating sulphur status with yields of

Phalaris tuberosa L. in the greenhouse. The highest correlations were with Ensminger's (1954) 500 ppm phosphate solution (r = 0.86) and with the Aspergillus niger microbiological assay (r = 0.85). They suggested that the superiority of these methods lies in the ability of the phosphate ions to replace adsorbed sulphate and the micro-organism's ability to utilize adsorbed sulphate.

Lowe (1963) showed an increase in the phosphate ion concentration over the range 0 to 0.5 M increased the amount of extractable sulphate. He also suggested that the pH of the phosphate solution be adjusted to pH 7.0.

McClung and de Freitas et al. (1959) found a high correlation between an ammonium acetate extraction and plant yield while Spencer and Freney (1960) found a moderately low correlation for the same extraction. Ensminger (1954) found little difference between ammonium acetate and potassium phosphate as extractants. Williams and Steinbergs (1962) found that a fraction designated as "heat soluble sulphur" had a higher "r" value with plant growth than other measures of available sulphur. Kilmer and Nearpass (1960) found the fraction extracted by 0.5 M sodium bicarbonate at pH 8.5 correlated well with sulphur "A" values. They concluded that this reagent extracts a fraction of the organic as well as the inorganic sulphate. Larger amounts of sulphate are extracted with sodium bicarbonate at pH 10 than at pH 8.5 (Kilmer and Nearpass, 1960) due to the increased solubility of sulphur-containing organic matter. Harward et al. (1962b) found no relationship between organic sulphate and plant yield.

Water soluble sulphate is generally a poor index of crop response (Walker, 1959; Williams and Steinbergs, 1959; Spencer and Freney, 1960). Ensminger (1954) found water extracted practically no sulphate.

Bardsley and Lancaster (1960) found a correlation of r = 0.790

between reserve sulphur (total soil sulphur minus soluble sulphate-sulphur) and yield of sulphur from 3 harvests of white clover but did not determine the relationship between reserve sulphur and plant yield. Lowe (1965) suggested the hydriodic-reducible sulphur may be a good indicator of the sulphur status of the soil. Spencer and Freney (1960) previously investigated the same fraction and reported a correlation of 0.753 between "reducible sulphur" and crop growth. They found the values for the reducible sulphur were considerably higher than plant uptake of sulphur. This fraction formed one-third to one-half of the total sulphur in the soils investigated. It includes most of the inorganic sulphur plus organic sulphates. According to Freney (1958) it comprises a larger portion of the organic sulphate than does heat soluble sulphur.

The basic weakness of using extractants as indicators of sulphur status is their failure to measure reserve sulphur or any form of it.

It is now well established that crops, especially the crops requiring large amounts of sulphur, contain less soluble sulphate in the herbage if grown on sulphur-deficient soil (Spencer, 1959; Dijshoorn et al., 1960; Walker and Bentley, 1961; Jones, 1962, 1963; Jones and Martin, 1964). It is not possible to determine the degree of sulphur deficiency from plant extraction methods. Walker and Bentley (1961) found 70 per cent hot ethanol extracted amounts of sulphate which were highly related to the sulphur status of the soil, but could not determine the degree of sulphur deficiency. The critical level of sulphate in alfalfa was found to be 0.09 per cent by Walker and Bentley (1961). A fraction, referred to as "extractable sulphur" by the same authors, was equally successful in assessing the sulphur status of the soil. Jones (1962) found the critical sulphate concentration in subclover to be 0.017 per cent.

Walker (1959) found no relationship between total nitrogen or amide nitrogen and legume yield, while Thomas (1950b) reported higher amide nitrogen concentrations in plants grown on soils deficient in sulphur. Dijshoorn et al. (1960) suggests that plants are sulphur-deficient when total sulphur content is less than 0.027 X Kjeldahl nitrogen.

Aspergillus niger has been found to be a simple and effective method for estimating the sulphur status of the soil (Spencer and Freney, 1960). Walker (1959), however, did not find the Aspergillus niger method to be effective for Alberta soils.

## Summary of the Literature Review

1. Sulphur is an essential plant nutrient required in substantial amounts.

2. Sulphur-deficient plants become chlorotic and growth is reduced.

3. Soils contain variable amounts of sulphur. Many soils in widespread areas of the world are sulphur-deficient for normal plant usage.

4. Losses of sulphur from the soil occur by leaching, erosion, and crop removal.

5. Sulphur is added to the soil through rainwater, especially near industrial centers, atmosphere contamination, manures, insecticides, fungicides, and fertilizers.

6. Some soils have the ability to retain sulphate by a mechanism of "anion exchange", however other mechanisms appear to be involved.

7. There is no adequate method of predicting sulphur-deficiency in the soil. Phosphate extractions have given the highest correlations with crop yield.

8. Legumes grown on sulphur-deficient soil contain lower amounts of inorganic sulphur than plants grown on sulphur-sufficient soil.

# MATERIAL AND METHODS

## A. Field Plots

In 1963 and 1964, 142 test locations were chosen on predominantly Grey Wooded soils of Alberta with the assistance of the District Agriculturalist or the Field Supervisor of the districts studied. Nine of the plots were abandoned and are not included in further discussion. The sites were located in an area between Whitecourt on the west and Bonnyville on the east, Athabasca on the north and Looma on the south. The area and the sites are shown in Figure 1.

In the selection of the test sites the following criteria were sought and in most cases met.

1. Soils were Dark Grey, Dark Grey Wooded, and Grey Wooded on well drained or moderately well drained sites. The topography was uniform and depressional areas were avoided.

2. Sites were chosen where sulphur-containing fertilizers had not been used for at least 3 years. In most cases these fertilizers had never been used.

3. Forage stands, consisting of at least 25 per cent legume, were chosen as the indicator crop. Poor growth of legume did not eliminate the site, but a uniform stand was sought.

4. Pasture fields where livestock would be present were avoided.

5. The locations were distributed as randomly as possible throughout the district.

The soils were classified by the Alberta Soil Survey[1]. The majority of the soils were in the Grey Wooded and Dark Grey Soil Zones;

[1] Personal communication with A. A. Kjearsgaard.

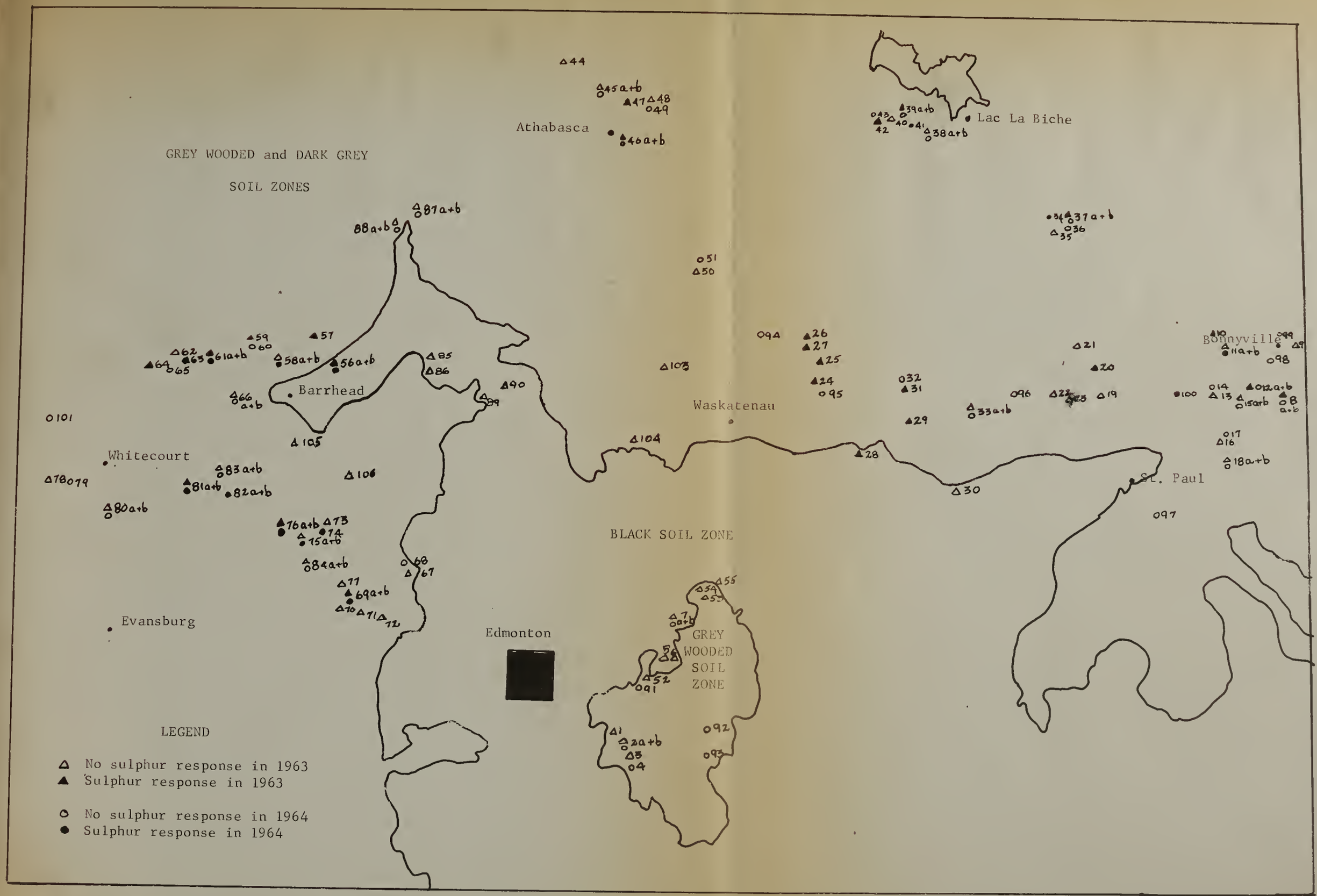



Figure 1. Map of area studied showing test sites. For complete description of sites see Table 1.

however, a few were in the Black Soil Zone. The site locations and classification of the soils are presented in Table 1.

In May of each year sodium sulphate was applied to the plot area at the rate of 88 lb./acre (20 lb. sulphur, 10 ppm in the surface 6 inches). The 1963 plot consisted of a fertilized area 2 rods square surrounded by an unfertilized border. In 1964, four treatments, each on an area 2 rods square were applied, viz: unfertilized, S, NPK, NPKS. The NPK was obtained from an application of 150 lb. 11-48-0/acre and 60 lb. 0-0-60 / acre. All applications were made with a fertilizer attachment mounted between 2 bicycle wheels.

Soil samples for laboratory analyses were taken from each site at 0-6 inch, 6-12 inch, 12-24 inch, 24-36 inch, and 36-48 inch depths with a Bull core sampler[1]. Each sample was a composite of four cores, one taken at each corner of the plot.

Rain gauges[2] were placed at the center of most experimental sites. A small amount of mineral oil was placed in each rain gauge to prevent evaporation.

During the growing season periodic observations were made of each plot for response of the legume to the applied sulphur and rainfall was recorded. The same observations were made at harvest time. If a response to the applied nutrient was observed in either the first or second cutting, the soil was considered sulphur-deficient.

The alfalfa stands were harvested as near to 10 per cent legume bloom as possible. Red clover and alsike clover were harvested at 100 per cent bloom. Two square-yard samples of plant material were taken from each treatment in a selected manner. The total sample was taken to the laboratory, dried and weighed.

[1] Product of A.D. Bull Enterprises, Chickasha, Oklahoma, U.S.A.
[2] Product of Victor Rain Gauge Co., Berryville, Arkansas, U.S.A.

Table 1. Location and Classification of Soils Studied

| Site | Cooperator | Legal Location | Soil Order | Sub-group | Soil Series | Parent Material |
|---|---|---|---|---|---|---|
| 1 | A. Ceretzke | NW 30-51-22-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 2* | A. Portas | NW 21-51-22-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 3 | F. Gibson | NW 11-51-22-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 4 | F. Gibson | NW 11-51-22-W4 | Podzolic | Grey Wooded | Maywood | Glacial till |
| 5 | S. Kinch | SE 22-53-21-W4 | Chernozemic | Dark Grey | Mico | Lacustrine |
| 6 | J. Corcoran | NW 23-53-21-W4 | Chernozemic | Dark Grey | Mico | Lacustrine |
| 7* | D. Bushnell | NE 11-54-21-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 8* | F. Besaraba | NW 21-60- 5-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 9 | A. LaForce | NE 21-61- 5-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 10 | C. Makaruk | NE 30-61- 7-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 11* | M. Selezinka | SE 29-61- 7-W4 | Podzolic | Grey Wooded | Culp | Glacial till |
| 12* | L. Kissell | NE 16-60- 6-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 13 | D. Ross | SW 17-60-77-W4 | Chernozemic | Dark Grey | Rimbey | Glacial till |
| 14 | D. Ross | NW 17-60- 7-W4 | Chernozemic | Dark Grey | Rimbey | Glacial till |
| 15* | A. Ritchie | SW 6-60- 6-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 16 | H. Elrick | SW 4-59- 7-W4 | Podzolic | Dark Grey | Uncas | Glacial till |
| 17 | H. Elrick | SW 4-59- 7-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |

* Site had tests in 1963 and 1964 and these are subsequently referred to as "a" and "b", respectively. e.g. 8a refers to 1963 site.

Table 1. (Continued)

| Site | Cooperator | Legal Location | Soil Order | Sub-group | Soil Series | Parent Material |
|---|---|---|---|---|---|---|
| 18* | F.Zacharuk | NE 10-58- 7-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 19 | R.Sloan | SW 3-60-10-W4 | Podzolic | Dark Grey | Uncas | Glacial till |
| 20 | J.Dallaire | SE 34-60-10-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 21 | K.Midwinter | SE 13-62-11-W4 | Podzolic | Dark Grey | Uncas | Glacial till |
| 22 | A.Krevinky | NE 4-61-11-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 23 | E.Murray | NW 3-60- 7-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 24 | W.Konasiuwich | NE 17-60-17-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 25 | J.Jusypink | SW 33-60-17-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 26 | J.Korbet | NE 19-61-17-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 27 | W.Holowaychuk | NE 7-61-17-W4 | Podzolic | Dark Grey | Uncas | Glacial till |
| 28 | J.Rubuliak | SW 28-58-16-W4 | Chernozemic | Dark Grey | Redwater | Alluvial aeolian |
| 29 | F.Tannis | NE 10-60-16-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 30 | A.Dary | SE 23-57-14-W4 | -- | -- | -- | -- |
| 31 | K.Charchuk | SE 10-60-15-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 32 | K.Charchuk | NE 10-60-15-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 33* | H.Sharlicki | SW 33-59-13-W4 | Podzolic | Grey Wooded | Uncas | Glacial till |
| 34 | G.Parker | SE 28-64-11-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |

Table 1. (Continued)

| Site | Cooperator | Legal Location | Soil Order | Sub-group | Soil Series | Parent Material |
|---|---|---|---|---|---|---|
| 35 | J.Andrichuk | SE 15-64-11-W4 | Podzolic | Grey Wooded | Maywood | Lacustrine |
| 36 | J.Andrichuk | SW 14-64-11-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 37* | G.Andrichuk | SE 27-64-11-W4 | Podzolic | Grey Wooded | Culp | Alluvial aeolian |
| 38* | L.L'Heureux | SW 31-66-14-W4 | Podzolic | Dark Grey | Uncas | Glacial till |
| 39* | A.Girard | SW 10-67-15-W4 | Podzolic | Grey Wooded | -- | -- |
| 40 | J.L'Heureux | SW 4-67-15-W4 | Chernozemic | Grey Wooded | Falun | Glacial till |
| 41 | J.L'Heureux | SW 34-66-15-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 42 | D.Amiot | SE 5-67-15-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 43 | D.Amiot | SE 5-67-15-W4 | Podzolic | Grey Wooded | -- | Glacial till |
| 44 | W.Bosik | SW 8-68-23-W4 | Podzolic | Grey Wooded | Tollman | Alluvial lacustrine |
| 45* | S.Zachoda | SW 18-67-22-W4 | Gleysolic | Humic Eluviated Gleysol | -- | Glacial till |
| 46* | M.Breckenridge | SE 26-66-22-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 47 | T.Salé | NE 18-67-21-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 48 | S.Chorzemka | NE 18-67-21-W4 | Gleysolic | Humic Gleysol | -- | -- |
| 49 | S.Chorzemka | NE 18-67-21-W4 | Gleysolic | Humic Eluviated Gleysol | -- | Lacustrine |
| 50 | G.Madden | SW 3-64-20-W4 | Podzolic | Grey Wooded | Uncas | Glacial till |
| 51 | G.Madden | SW 3-64-20-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |

Table 1. (Continued)

| Site | Cooperator | Legal Location | Soil Order | Sub-group | Soil Series | Parent Material |
|---|---|---|---|---|---|---|
| 52 | J.Andrew | SE 7-53-21-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 53 | G.Walkowski | NW 3-55-20-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 54 | V.Schneider | NW 9-55-20-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 55 | S.Stelter | NW 13-55-20-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 56* | O.Messmer | NE 19-60- 2-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 57 | P.Mast | SW 3-61- 3-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 58* | C.Pusch | SE 25-60- 4-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 59 | J.Rattray | SE 31-60- 4-W5 | Podzolic | Grey Wooded | Tollman | Alluvial lacustrine |
| 60 | J.Rattray | SE 31-60- 4-W5 | Gleysolic | Low Humic Eluviated Gleysol | -- | Alluvial lacustrine |
| 61* | F.Pearson | NW 19-60- 5-W5 | Gleysolic | Humic Eluviated Gleysol | -- | Alluvial lacustrine |
| 62 | K.MacKenzie | SE 22-60- 6-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 63 | K.MacKenzie | NW 13-60- 6-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 64 | F.Harrison | SE 1-60- 7-W5 | Podzolic | Grey Wooded | -- | Alluvial lacustrine |
| 65 | F.Harrison | SW 6-60- 6-W5 | Podzolic | Grey Wooded | -- | Glacial till |
| 66* | F.Kinnaird | SW 26-59- 5-W5 | Podzolic | Grey Wooded | -- | Glacial till |
| 67 | P.McLeod | SW 16-55-27-W4 | Solonetzic | Dk.Grey Solodized Solonetz | Thorsby | -- |
| 68 | P.McLeod | SW 16-55-27-W4 | Solonetzic | Grey Wooded Solodized Solonetz | Kawood | Glacial till |
| 69* | T.Smith | NE 36-54- 2-W5 | Podzolic | Grey Wooded | Leith | Glacial till |

Table 1. (Continued)

| Site | Cooperator | Legal Location | Soil Order | Sub-group | Soil Series | Parent Material |
|---|---|---|---|---|---|---|
| 70 | A.Salter | NE 26-54- 2-W5 | Podzolic | Grey Wooded | Uncas | Glacial till |
| 71 | E.Phillips | NE 19-54- 1-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 72 | C.Meyer | NW 21-54- 1-W5 | Chernozemic | Dark Grey | Rimbey | Glacial till |
| 73 | G.Robatham | SE 18-56- 2-W5 | Chernozemic | Dark Grey | Mico | Glacial till |
| 74 | G.Robatham | SE 18-56- 2-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 75* | C.Brand | SW 11-56- 3-W5 | Podzolic | Grey Wooded | Uncas | Glacial till |
| 76* | C.Wood | SE 18-56- 3-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 77 | A.Foht | SW 12-55- 2-W5 | Podzolic | Grey Wooded | Maywood | Lacustrine |
| 78 | R.Commandeur | NE 8-57- 9-W5 | Podzolic | Grey Wooded | -- | Lacustrine |
| 79 | R.Commandeur | SW 9-57- 9-W5 | Podzolic | Grey Wooded | -- | Glacial till |
| 80* | J.Holland | NW 23-56- 8-W5 | Podzolic | Grey Wooded | Maywood | Lacustrine |
| 81* | I.Galbraith | NE 10-57- 6-W5 | Podzolic | Grey Wooded | -- | Alluvial lacustrine |
| 82* | P.Aasland | SW 10-57- 5-W5 | Podzolic | Grey Wooded | Maywood | Lacustrine |
| 83* | E.MacIntosh | NE 17-57- 5-W5 | Podzolic | Grey Wooded | Maywood | Lacustrine |
| 84* | P.Pastushak | NW 26-55- 4-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 85 | P.Letts | NW 22-60-27-W4 | Gleysolic | Humic Gleysol | -- | Glacial till |
| 86 | A.Lyons | SE 15-60-27-W4 | Gleysolic | Humic Eluviated Gleysol | -- | Glacial till |
| 87* | A.Watson | NE 12-64- 1-W5 | Gleysolic | Humic Gleysol | -- | Alluvial lacustrine |
| 88* | E.Marshall | SE 34-63- 1-W5 | Regosolic | Gleyed Regosolic | -- | Alluvial lacustrine |

Table 1. (Continued)

| Site | Cooperator | Legal Location | Soil Order | Sub-group | Soil Series | Plant Material |
|---|---|---|---|---|---|---|
| 89 | F.Pankonin | NW 25-59-26-W4 | Solonetzic | Solodized Solonetz | Kavanaugh | Residual |
| 90 | R.Shank | NE 2-60-25-W4 | Solonetzic | Solodized Solonetz | Kavanaugh | Residual |
| 91 | R.Parker | NE 1-53-22-W4 | Podzolic | Grey Wooded | Uncas | Glacial till |
| 92 | J.Dunn | NW 34-51-20-W4 | Podzolic | Grey Wooded | Uncas | Glacial till |
| 93 | V.Bates | NE 10-51-20-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 94 | Laukkanen Bros. | SW 12-61-19-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 95 | G.Elaschuk | NE 4-60-17-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 96 | W.Marceniuk | NW 17-60-12-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 97 | J.Saeger | NW 10-57- 9-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 98 | C.Dery | NE 24-61- 6-W4 | Chernozemic | Dark Grey | Falun | Glacial till |
| 99 | S.Baik | SW 6-61- 5-W4 | Podzolic | Dark Grey | Uncas | Glacial till |
| 100 | R.St.Arnault | NW 8-60- 9-W4 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 101 | Mr. Selleck | NE 35-58- 7-W5 | Podzolic | Grey Wooded | -- | Alluvial aeolian |
| 102 | R.Livermore | NW 3-55- 4-W5 | Podzolic | Grey Wooded | Cooking Lake | Glacial till |
| 103 | F.Bolton | NE 26-60-21-W4 | -- | -- | -- | -- |
| 104 | R. Kondro | SW 36-58-22-W4 | -- | -- | -- | -- |
| 105 | W.Rendfleisch | NE 17-58- 3-W5 | -- | -- | -- | -- |
| 106 | J.Redwood | SW 23-57- 2-W5 | -- | -- | -- | -- |

* Site had tests in 1963 and 1964 and these are subsequently referred to as "a" and "b", respectively. e.g. 8a refers to 1963 site.

Botanically pure samples of alfalfa were taken from the non-flowering terminal portion of plants in blossom to assure equal physiological age. For red clover and alsike clover the bloom was included in the sample. Some 100 to 125 g. of plant material (green weight) were collected at random to give approximately 25 g. of dry material. The samples were immediately transferred to a refrigerated container and frozen.

## B. Laboratory Analyses

### (1) Plant Samples

The plant samples were stored in a deepfreeze until they were dried in a freeze dryer. Following drying the samples were ground in a Wiley (intermediate model) laboratory grinding mill and stored in labelled glass containers until analyzed.

Ammoniacal and organic nitrogen were determined on all plant samples by the Kjeldahl-Wilfarth-Gunning method (A.O.A.C., 1955). A commercial catalyst containing $K_2SO_4$, HgO, and $CuSO_4$ was used. The ammonia was collected in 4 per cent boric acid as suggested by Meeker and Wagner (1933) and titrated against standardized sulphuric acid. Soluble sulphate, extracted by hot 70 per cent ethanol, was determined by the method of Walker (1961).

### (2) Soil Samples

The soil samples were air dried and ground to pass through a 2 mm. sieve. A separate soil moisture sample previously collected and weighed in the field, was oven dried at 105° C. for 24 hours, weighed and the moisture percentage calculated. The 1/3 atmosphere method for field capacity (U.S.D.A. Handbook 60, 1954) was used on the soil samples collected in 1963.

Routine analyses were done by the Agricultural Soil and Feed Testing Laboratory on the 0-6 inch and 6-12 inch samples for all test sites. Nitrate nitrogen was determined as outlined by Spurway and Lawton (1949). To 5 g. of soil were added 25 ml. of 0.024 N HAc, then shaken for 2 minutes and filtered. The nitrate was determined with diphenylamine solution. The Kitson and Mellon (1944) method was used to determine phosphorus. After extracting with 0.03 N $H_2SO_4$ and 0.03 N $NH_4F$, the color was developed by adding 5 ml. each of 5 N sulphuric acid, 0.25 per cent ammonium vanadate, and 5 per cent ammonium molybdate in succession to the extract and reading in a spectrophotometer. Available potassium was estimated by the Spurway and Lawton procedure (1949). The potassium in the 0.024 N HAc extract was determined on a Baird flame photometer.

The soil paste method of Doughty (1942) was used to determine the pH of the soil samples. A Beckman H-2 pH meter equipped with glass and calomel electrodes was used.

For a more comprehensive study of the soils, 4 responsive soils and 4 non-responsive soils were chosen. For the purpose of this study, a responsive soil was one on which legumes responded to applied sulphur and a non-responsive soil was one on which legumes did not respond to applied sulphur. The following were the criteria for selection of the 8 soils:

1. The plant yields were substantial.

2. The same behavior was noted on the plots for both years of the study.

3. The duplicate sites were close together both years.

4. Separation of responsive and non-responsive soils could be made by the hot 70 per cent ethanol extractable method of Walker and Bentley (1961).

5. There was a range of available phosphate.

6. The soils were in the Podzolic or Gleysolic orders.

The eight soils studied are listed in Table 2.

For the comprehensive study, composite soil samples for each depth at each site were prepared by mixing equal weights of the sample obtained in each of the two years. A portion of the bulked sample was ground with a mortar and pestle to pass through a 60 mesh sieve.

Total carbon was determined on the Leco Induction Furnace (Leco Equipment Corporation, 1957). Organic and ammoniacal nitrogen were determined on the selected soil samples by the Kjeldahl method referred to previously. Mechanical analyses was preformed by the method outlined by Toogood and Peters (1953).

Total sulphur was determined on the Leco Induction Furnace and Titrator (Leco Equipment Corporation, 1959). A 0.25 g. sample of 60 mesh soil was placed in a crucible with: a 0.5 g. copper ring, approximately 2.5 g. powdered sulphur-free iron, and 1.0 g. of powdered tin. The $SO_2$ driven off was titrated against a standard iodate solution. The blank often represented 80 to 90 per cent of the final reading. For this reason precision was low with error ranging from 15 to 25 per cent. The copper ring was responsible for the high blank, but it was necessary to obtain acceptable results. The method has the advantage of being rapid.

Several sulphur fractions were determined. All of the final estimations of sulphur throughout the study in the extractions were made by the Johnson and Nishita method (1952). The essential features of the method included reduction of the sulphur-containing compounds to sulphide by a boiling hydryiodic and hypophosphorous acid mixture, trapping the liberated sulphide in zinc acetate and development of the methylene blue

Table 2. Soils Used in Detailed Study

| Sample | Site | Depth in Inches | Soil Series |
|---|---|---|---|
| I. Non-responsive sites: | | | |
| A1 | 45 | 0- 6 | * |
| A2 | | 6-12 | |
| A3 | | 12-24 | |
| A4 | | 24-36 | |
| B1 | 66 | 0- 6 | ** |
| B2 | | 6-12 | |
| B3 | | 12-24 | |
| B4 | | 24-36 | |
| C1 | 80 | 0- 6 | Maywood Si. C. |
| C2 | | 6-12 | |
| C3 | | 12-24 | |
| C4 | | 24-36 | |
| D1 | 84 | 0- 6 | Cooking Lake Si. L. |
| D2 | | 6-12 | |
| D3 | | 12-24 | |
| D4 | | 24-36 | |
| II. Responsive sites: | | | |
| E1 | 76 | 0- 6 | Cooking Lake Si. L. |
| E2 | | 6-12 | |
| E3 | | 12-24 | |
| E4 | | 24-36 | |
| F1 | 69 | 0- 6 | Leith S. L. |
| F2 | | 6-12 | |
| F3 | | 12-24 | |
| F4 | | 24-36 | |
| G1 | 46 | 0- 6 | Cooking Lake L. |
| G2 | | 6-12 | |
| G3 | | 12-24 | |
| G4 | | 24-36 | |
| H1 | 56 | 0- 6 | Cooking Lake Si. L. |
| H2 | | 6-12 | |
| H3 | | 12-24 | |
| H4 | | 24-36 | |

* Not named, was a Humic Eluviated Gleysol.
** Not named, was an Orthic Grey Wooded.

color with N-N-dimethyl-p-phenylenediaminesulphate.. The color intensity was read in a Beckman model B spectrophotometer.

One hundred ml. digestion flasks were used. The flask was attached to a condenser which was connected to a washing column. The collecting units were 50 ml. volumetric flasks. Two ml. of sample and 4 ml. of reducing solution were used except in the following cases. Slight modifications were made in the method for the very low concentrations of sulphur when a 4 ml. aliquot of sample and 8 ml. of reducing solution were used. The sulphide was trapped in a 25 ml. volumetric flask. The method had high precision at low concentrations of sulphur. The accuracy is higher than for other methods reported in the literature.

Two problems were encountered. The nitrogen flow rate proved to be critical on the apparatus used in this study. If the flow rate was too high hydriodic acid was carried over into the zinc acetate trapping solution causing a brown color to form instead of the characteristic blue of methylene blue. Contamination from the atmosphere was encountered when the apparatus was left open at night. The error was corrected when the system remained closed.

The HI-reducible sulphur fraction was determined by the method of Spencer and Freney (1960). A 0.100 to 0.500 g. soil sample was put directly into the digestion flask of the Johnson and Nishita apparatus and 4 ml. of reducing mixture was added. After heating for 1 hour the methylene blue color was developed. Error for this method was about 5 per cent.

Carbon bonded sulphur was determined by the method of DeLong and Lowe (1962). A 0.100 to 0.500 soil sample was placed in the digestion flask of Johnson and Nishita (1952). The other essential features were desulphurization of organic compounds with Raney nickel in a basic media, acidification with HCl and development of methylene blue.

The 500 ppm phosphate extractable sulphate was determined by the method of Ensminger (1954) and is hereafter referred to as "extractable sulphate". A 20.0 g. soil sample was equilibrated with 100 ml. of $KH_2PO_4$ (500 ppm P) for one-half hour. After extraction by filtration the sulphate was determined using the Johnson and Nishita method (1952). The extract contained water soluble as well as adsorbed sulphate. Good precision was obtained between the duplicate soil extractions.

Easily soluble and adsorbed sulphate were determined by Lowe's method (1965). Duplicate 20.0 g. soil samples were shaken for 20 minutes in 50 ml. of 0.001 N HCl, then leached with an additional 45 ml. acid. The combined extract was made up to a volume of 100 ml. The same sample was leached with 90 ml. of 0.5 M phosphate solution buffered at pH 7.0. The leachate was made up to 100 ml. volume. The sulphate in the 2 extracts was determined by the methylene blue method (Johnson and Nishita, 1952).

# RESULTS AND DISCUSSION

## A. Field Plots

The sulphur status of the soil was evaluated on the basis of response of the legumes to applied sulphur. Responses were defined on the basis of visual observation and yield data with the main emphasis on the former. The duplicate square yard determinations frequently varied by 50 per cent and these differences were often greater than differences between plots. If the crop response to sulphur was dubious, as it was in many cases, the soil was included in the non-responsive category. If, however, the crop grown on the soil responded to sulphur application either in the first or second cut, it was put in the responsive category. Some plots were still difficult to separate. There were many errors inherent in this method of evaluating the sulphur status of the soil, but it was the best alternative available. Controlled conditions are difficult to obtain in the field. The results, therefore, apply only to the years studied. Soils which were found to be non-responsive in this investigation may respond in years of higher rainfall or if cropping intensity is increased. The errors, in the author's opinion, resulted in an underestimation rather than overestimation of the extent of sulphur deficiency.

Tables 3 and 4 give the field data for 1963 and 1964. Response to the applied sulphur occurred on 28 of 78 soils in 1963 and on 16 of 55 soils in 1964. Thus, 31 per cent of the sites investigated in this study were sulphur-responsive, compared to 45 per cent for the area studied by Walker (1959).

Two of the sites studied (29 and 94) were located on the same quarter-sections as 2 plots previously investigated by Hoff (1953). In both cases site 29 was responsive and site 94 was non-responsive.

Table 3. Dry Matter Yields, Available Nutrients, and pH of 1963 Sites

| Site | Series | Predominant Legume | Yields, Tons/ac. Check Plot | S. Plot | Visual Response | Available Nutrients pp2m N | P | K | pH |
|---|---|---|---|---|---|---|---|---|---|
| 1 | Ck.L. | Alfalfa | 0.43 | 0.44 | No | 11 | 52 | 178 | 6.1 |
| 2a | Ck.L. | Alfalfa | 1.22 | 1.48 | No | 2 | 14 | 98 | 6.2 |
| 3 | Ck.L. | Alfalfa | 1.03 | 1.55 | No | 9 | 22 | 88 | 6.0 |
| 5 | Mc. | Alfalfa | 1.26 | 0.84 | No | 4 | 3 | 68 | 4.9 |
| 6 | Mc. | Alfalfa | 0.68 | 0.64 | No | 2 | 3 | 58 | 5.3 |
| 7a | Fn. | Alfalfa | 0.86 | 1.25 | No | 2 | 7 | 78 | 6.5 |
| 8a | Ck.L. | Alfalfa | 0.65 | 1.06 | Yes | 2 | 42 | 78 | 6.7 |
| 9 | Fn. | Alfalfa | 1.03 | 1.12 | No | 11 | 8 | 36 | 7.2 |
| 10 | Ck.L. | Alfalfa | 0.42 | 0.72 | Yes | 4 | 42 | 52 | 6.8 |
| 11 | Cu. | Alfalfa | -- | -- | No | 2 | 124 | 60 | 6.9 |
| 12a | Ck.L. | Alfalfa | 1.09 | 1.23 | Yes** | 2 | 77 | 60 | 7.8 |
| 13 | Rm. | Alfalfa | -- | -- | No | 9 | 8 | 36 | 6.3 |
| 15a | Ck.L. | Alfalfa | 1.38 | 1.40 | No | 4 | 19 | 82 | 6.2 |
| 16 | Un. | Alfalfa | 0.65 | 0.68 | No | 7 | 3 | 26 | 6.8 |
| 18a | Ck.L. | Alfalfa | 0.73 | 0.58 | No | 7 | 17 | 32 | 6.3 |
| 19 | Un. | Alfalfa | -- | -- | No | -- | 16 | 40 | 6.4 |
| 20 | Ck.L. | Alfalfa | 0.79 | 1.20 | Yes | 2 | 108 | 118 | 6.9 |
| 21 | Un. | Alfalfa | -- | -- | No | 2 | 9 | 34 | 6.2 |
| 22 | Ck.L. | Alfalfa | 1.21 | 1.33 | No | 2 | 16 | 50 | 6.2 |
| 23 | Ck.L. | Alfalfa | 0.61 | 0.55 | No | 2 | 8 | 34 | 5.8 |
| 24 | Ck.L. | Alfalfa | 0.81 | 1.01 | Yes | 0 | 229 | 174 | 6.9 |
| 25 | Ck.L. | Alfalfa | 1.02 | 1.46 | Yes | 0 | 55 | 60 | 6.6 |
| 26 | Ck.L. | Alfalfa | 0.80 | 1.07 | Yes | 2 | 21 | 88 | 6.6 |
| 27 | Un. | Alfalfa | 0.94 | 1.36 | Yes | 2 | 98 | 66 | 7.1 |
| 28 | Rw. | Alfalfa | 0.72 | 0.98 | Yes | 0 | 84 | 120 | 6.8 |
| 29 | Ck.L. | Alfalfa | 0.29 | 0.63 | Yes | 0 | 34 | 62 | 6.7 |
| 30 | -- | Alfalfa | 0.64 | 0.60 | No | 4 | 236 | 116 | 6.3 |
| 31 | Ck.L. | Alfalfa | 1.05 | 1.59 | Yes | 7 | 12 | 58 | 6.1 |
| 33a | Un. | Alfalfa | -- | -- | No | 13 | 8 | 32 | 6.1 |
| 35 | Ck.L. | Alfalfa | -- | -- | No | 18 | 20 | 52 | 6.7 |
| 37a | Cu. | Alfalfa | 1.03 | 1.89 | Yes | 4 | 139 | 94 | 6.5 |
| 38a | Un. | Alfalfa | 2.17 | 2.45 | No | 18 | 14 | 54 | 7.0 |
| 39a | * | Alsike | 0.92 | 1.01 | Yes | 18 | 19 | 52 | 6.5 |
| 40 | Fn. | Red clover | 1.25 | 1.40 | No | 13 | 17 | 60 | 6.1 |
| 42 | Fn. | Alfalfa | 1.12 | 1.29 | Yes | 31 | 4 | 70 | 6.4 |
| 44 | To. | Alfalfa | 1.02 | 1.10 | No | 4 | 98 | 104 | 7.5 |

* Series not named, for complete information see Table 1.
** Response observed in the second cut only.
T Trace.

Table 3. (Continued)

| Site | Series | Predominant Legume | Yields, Tons/ac. Check Plot | Yields, Tons/ac. S. Plot | Visual Response | Available Nutrients pp2m N | P | K | pH |
|---|---|---|---|---|---|---|---|---|---|
| 45a | * | Alsike | 1.10 | 1.45 | No | 13 | 7 | 76 | 7.1 |
| 46a | Ck.L. | Alfalfa | 0.67 | 1.02 | Yes | 2 | 26 | 74 | 6.5 |
| 47 | Ck.L. | Alfalfa | 0.22 | 0.62 | Yes | 2 | 93 | 52 | 6.4 |
| 48 | * | Alfalfa | 1.42 | 1.52 | No | T | 4 | 50 | 7.8 |
| 50 | Un. | Red clover | 1.55 | 1.37 | No | T | 15 | 46 | 6.0 |
| 52 | Fn. | Alfalfa | 0.49 | 0.94 | No | 7 | 6 | 114 | 6.5 |
| 53 | Fn. | Alfalfa | 1.38 | 2.00 | No | 11 | 11 | 36 | 7.0 |
| 54 | Ck.L. | Alfalfa | 1.15 | 1.21 | No | 7 | 90 | 144 | 6.8 |
| 55 | Ck.L. | Alfalfa | 1.96 | 1.76 | No | 13 | 52 | 66 | 6.6 |
| 56a | Ck.L. | Alfalfa | 1.08 | 1.41 | Yes | 9 | 39 | 68 | 6.5 |
| 57 | Ck.L. | Alfalfa | 1.43 | 1.61 | Yes | 9 | 165 | 120 | 6.8 |
| 58 | Ck.L. | Alfalfa | 1.12 | 1.52 | No | 13 | 115 | 82 | 7.1 |
| 59 | To. | Red clover | 0.80 | 1.11 | Yes | 11 | 35 | 60 | 6.9 |
| 61a | * | Alfalfa | 1.13 | 1.19 | Yes | 2 | 21 | 68 | 7.4 |
| 62 | Ck.L. | Alfalfa | 1.32 | 1.52 | No | 2 | 20 | 74 | 6.4 |
| 64 | * | Alsike | 1.31 | 2.00 | Yes | 11 | 67 | 196 | 7.5 |
| 66a | * | Alfalfa | 0.98 | 1.46 | No | 4 | 16 | 48 | 6.8 |
| 67 | To. | Alfalfa | 0.69 | 0.87 | No | 7 | 10 | 30 | 5.5 |
| 69a | Le. | Alfalfa | 0.85 | 1.32 | Yes | 7 | 143 | 86 | 6.5 |
| 70 | Un. | Alfalfa | 0.36 | 0.33 | No | 7 | 68 | 40 | 6.3 |
| 71 | Ck.L. | Alfalfa | -- | -- | No | 9 | 96 | 102 | 6.6 |
| 72 | Rm. | Alfalfa | -- | -- | No | 11 | 9 | 34 | 6.6 |
| 73 | Mc. | Alfalfa | 0.97 | 0.98 | No | 7 | 19 | 36 | 5.9 |
| 75a | Un. | Alfalfa | 0.78 | 0.89 | No | 4 | 38 | 48 | 6.7 |
| 76a | Ck.L. | Alfalfa | 0.56 | 0.66 | Yes | 11 | 181 | 132 | 7.1 |
| 77 | Mw. | Alfalfa | -- | -- | No | 7 | 77 | 82 | 6.5 |
| 78 | * | Alfalfa | 1.07 | 1.15 | No | 7 | 8 | 66 | 6.2 |
| 80a | Mw. | Alsike | 1.12 | 1.23 | No | 11 | 3 | 68 | 5.8 |
| 81a | * | Alfalfa | 1.50 | 1.54 | Yes | 4 | 112 | 110 | 6.3 |
| 82a | Mw. | Alfalfa | 0.64 | 0.72 | Yes | 4 | 15 | 54 | 6.4 |
| 83a | Mw. | Alfalfa | 0.89 | 1.10 | No | 4 | 61 | 172 | 6.8 |
| 84a | Ck.L. | Alfalfa | 0.89 | 0.81 | No | 7 | 30 | 54 | 6.8 |
| 85 | * | Alfalfa | -- | -- | No | 9 | 4 | 22 | 7.8 |
| 86 | * | Alfalfa | 0.86 | 0.92 | No | 7 | 14 | 22 | 6.2 |
| 87a | * | Alfalfa | 1.85 | 2.00 | No | 7 | 23 | 160 | 7.8 |
| 88a | * | Alsike | 1.19 | 1.15 | No | 4 | 8 | 52 | 7.1 |

Table 3. (Continued)

| Site | Series | Predominant Legume | Yields, Tons/ac. Check Plot | Yields, Tons/ac. S. Plot | Visual Response | Available Nutrients pp2m N | P | K | pH |
|---|---|---|---|---|---|---|---|---|---|
| 89 | Kv. | Alfalfa | -- | -- | No | 4 | 47 | 32 | 5.8 |
| 90 | Kv. | Alfalfa | -- | -- | No | 11 | 14 | 12 | 6.1 |
| 103 | -- | Alfalfa | -- | -- | No | 0 | 14 | 50 | 7.0 |
| 104 | -- | Alfalfa | -- | -- | No | 0 | 14 | 58 | 7.7 |
| 105 | -- | Alfalfa | -- | -- | No | 4 | 42 | 42 | 6.5 |
| 106 | -- | Alfalfa | -- | -- | No | 7 | 6 | 36 | 6.5 |

* Series not named, for complete information see Table 1.

** Response observed in the second cut only.

T Trace.

Table 4

SEE OVER

Table 4. Dry Matter Yields, Available Nutrients, and pH of 1964 Plots

| Site | Soil Series | Predominant Legume | Check Plot Yield Tons/ac. | S. Plot Yield Tons/ac. | NPK Plot Yield Tons/ac. |
|---|---|---|---|---|---|
| 2b | Ck.L. | Alfalfa | 0.84 | 0.97 | 1.16 |
| 4 | Mw. | Alfalfa | 0.96 | 1.12 | 1.06 |
| 7b | Fn. | Alfalfa | 0.92 | 0.48 | 0.50 |
| 8b | Ck.L. | Alfalfa | -- | -- | -- |
| 11b | Cu. | Alfalfa | -- | -- | -- |
| 12b | Ck.L. | Alfalfa | -- | -- | -- |
| 14 | Rm. | Alfalfa | 0.64 | 0.43 | 0.68 |
| 15b | Ck.L. | Alfalfa | -- | -- | -- |
| 17 | Ck.L. | Alfalfa | 0.57 | 0.38 | 0.71 |
| 18b | Ck.L. | Alfalfa | 0.80 | 0.92 | 1.07 |
| 32 | Ck.L. | Alfalfa | 1.43 | 1.38 | 1.44 |
| 33b | Un. | Alfalfa | 0.31 | 0.39 | 0.29 |
| 34 | Ck.L. | Alfalfa | -- | -- | -- |
| 36 | Ck.L. | Alfalfa | 0.90 | 0.75 | 0.79 |
| 37b | Cu. | Alfalfa | 1.43 | 0.87 | 1.38 |
| 38b | Un. | Alfalfa | 1.35 | 1.07 | 1.21 |
| 39b | * | Alfalfa | 0.91 | 0.79 | 0.77 |
| 41 | Ck.L. | Alfalfa | 0.66 | 0.80 | 0.75 |
| 43 | * | Alfalfa | 1.17 | 0.88 | 1.31 |
| 45b | * | Alsike | 1.01 | 1.27 | 1.16 |
| 46b | Ck.L. | Alfalfa | 0.39 | 0.61 | 0.67 |
| 49 | * | Alfalfa | 1.15 | 1.36 | 1.08 |
| 51 | Ck.L. | Alsike | -- | -- | -- |
| 56b | Ck.L. | Alfalfa | 1.04 | 1.46 | 2.51 |
| 58b | Ck.L. | Alfalfa | 1.26 | 1.31 | 1.57 |
| 60 | * | Red clover | -- | -- | -- |
| 61b | * | Alfalfa | 1.16 | 0.81 | 1.02 |
| 63 | Ck.L. | Red clover | 0.63 | 0.99 | 1.13 |
| 65 | * | Alsike | 1.24 | 0.99 | 1.22 |
| 66b | * | Alfalfa | 1.06 | 1.24 | 1.46 |
| 68 | Kw. | Alfalfa | 0.75 | 0.71 | 0.96 |
| 69b | Le. | Alfalfa | 0.64 | 1.38 | 1.24 |

* Series not named, for complete information see Table 1.
** Response observed in the second cut only.

| NPKS Plot Yield Tons/ac. | Visual Response to S | Available Nutrients pp2m N | P | K | pH |
|---|---|---|---|---|---|
| 0.90 | No | 13 | 9 | 146 | 6.7 |
| 1.18 | No | 1 | 18 | 114 | 6.7 |
| 1.20 | No | 9 | 63 | 74 | 6.6 |
| -- | No | T | 52 | 60 | 6.8 |
| -- | Yes | 0 | 157 | 76 | 6.9 |
| -- | No | 0 | 49 | 94 | 7.7 |
| 0.64 | No | 2 | 7 | 58 | 6.5 |
| -- | No | 0 | 85 | 70 | 6.5 |
| 0.41 | No | T | 3 | 22 | 6.7 |
| 1.03 | No | 0 | 6 | 32 | 6.9 |
| 1.22 | No | 11 | 32 | 54 | 6.8 |
| 0.35 | No | 2 | 8 | 42 | 6.3 |
| -- | Yes | 0 | 41 | 46 | 6.9 |
| 0.82 | No | 0 | 20 | 48 | 6.2 |
| 1.27 | No | 0 | 189 | 76 | 6.3 |
| 1.18 | No | 0 | 23 | 46 | 7.1 |
| 0.78 | No | 0 | 36 | 94 | 6.6 |
| 1.12 | Yes | 0 | 232 | 106 | 7.0 |
| 0.92 | Yes** | 0 | 4 | 60 | 7.1 |
| 1.13 | No | 13 | 9 | 58 | 6.7 |
| 0.76 | Yes | 0 | 43 | 70 | 6.5 |
| 1.41 | No | 2 | 25 | 70 | 7.0 |
| -- | No | 31 | 33 | 60 | 6.6 |
| 1.92 | Yes | 7 | 51 | 52 | 6.5 |
| 1.55 | Yes** | 16 | 30 | 58 | 7.0 |
| -- | No | 16 | 2 | 50 | 8.2 |
| 1.11 | Yes** | 13 | 14 | 82 | 7.6 |
| 1.00 | Yes | T | 4 | 68 | 5.8 |
| 1.16 | No | 13 | 23 | 88 | 7.5 |
| 1.66 | No | 2 | 7 | 34 | 6.8 |
| 0.80 | No | 0 | 28 | 48 | 5.8 |
| 1.24 | Yes | 2 | 95 | 62 | 6.5 |

Table 4. (Continued)

SEE OVER

Table 4. (Continued)

| Site | Soil Series | Predominant Legume | Check Plot Yield Tons/ac. | S. Plot Yield Tons/ac. | NPK Plot Yield Tons/ac. |
|---|---|---|---|---|---|
| 74 | Ck.L. | Alfalfa | -- | -- | -- |
| 75b | Un. | Alfalfa | -- | -- | -- |
| 76b | Ck.L. | Alfalfa | 0.70 | 1.03 | 1.59 |
| 79 | * | Alfalfa | 0.56 | 0.71 | 0.62 |
| 80b | Mw. | Alsike | -- | -- | -- |
| 81b | * | Alfalfa | 1.78 | 1.68 | 2.27 |
| 82b | Mw. | Alfalfa | -- | -- | -- |
| 83b | Mw. | Alfalfa | -- | -- | -- |
| 84b | Ck.L. | Alfalfa | 1.64 | 1.55 | 1.78 |
| 87b | * | Alfalfa | 2.46 | 2.20 | 1.75 |
| 88b | * | Alsike | 0.51 | 0.52 | 1.12 |
| 91 | Un. | Alfalfa | 1.18 | 0.50 | 0.98 |
| 92 | Un. | Alfalfa | 1.16 | 1.40 | 1.46 |
| 93 | Fn. | Alfalfa | 0.96 | 1.12 | 1.06 |
| 94 | Ck.L. | Alfalfa | 0.98 | 0.98 | 1.00 |
| 95 | Fn. | Alfalfa | 1.31 | 1.44 | 1.48 |
| 96 | Ck.L. | Alfalfa | 0.95 | 1.03 | 0.92 |
| 97 | Ck.L. | Alfalfa | 0.99 | 0.90 | 0.90 |
| 98 | Fn. | Alfalfa | -- | -- | -- |
| 99 | Un. | Alfalfa | 0.67 | 0.96 | 0.63 |
| 100 | Ck.L. | Alfalfa | 0.48 | 0.90 | 0.47 |
| 101 | * | Alfalfa | 2.01 | 2.07 | 1.40 |
| 102 | Ck.L. | Red clover | 0.92 | 0.91 | 1.20 |

* Series not named, for complete information see Table 1.

| NPKS Plot Yield Tons/ac. | Visual Response to S | Available Nutrients pp2m N | P | K | pH |
|---|---|---|---|---|---|
| -- | Yes | 7 | 7 | 32 | 6.4 |
| -- | Yes | 9 | 25 | 58 | 6.7 |
| 1.14 | Yes | 2 | 142 | 104 | 7.0 |
| 0.88 | No | 0 | 15 | 120 | 6.5 |
| -- | No | T | 4 | 74 | 5.7 |
| 1.92 | Yes | 0 | 43 | 60 | 7.1 |
| -- | Yes | T | 14 | 46 | 6.4 |
| -- | No | 0 | 66 | 162 | 6.6 |
| 1.79 | No | 0 | 97 | 58 | 7.0 |
| 2.40 | No | 7 | 28 | 26 | 8.0 |
| 1.16 | No | 40 | 2 | 50 | 7.8 |
| 0.74 | No | 16 | 6 | 138 | 7.0 |
| 1.44 | No | T | 4 | 70 | 6.6 |
| 1.18 | No | T | 38 | 132 | 6.7 |
| 1.08 | No | 4 | 84 | 94 | 7.2 |
| 1.54 | No | T | 8 | 82 | 7.1 |
| 0.84 | No | 7 | 10 | 90 | 7.5 |
| 0.96 | No | 2 | 13 | 74 | 6.9 |
| -- | No | T | 22 | 116 | 6.1 |
| 0.75 | No | T | 87 | 34 | 7.1 |
| 1.19 | Yes | 2 | 61 | 26 | 6.9 |
| 1.92 | No | 16 | 61 | 98 | 6.4 |
| 1.20 | No | 0 | 0 | 62 | 6.8 |

Deficient soils were not restricted to particular geographic areas or to particular soil series. At 48 sites the soil used was classified as either Cooking Lake L. - S. L. or an Orthic Grey Wooded, both being developed on glacial till. Response was noted at 44 per cent of these sites compared to only 31 per cent for the entire project. Eighty-three per cent of the legume crops grown on the sandier soils responded to sulphur compared to 9 per cent for the finer textured soils. The yield increases also appeared to be greater on the sandy soils. However, the number of sites with sandy or fine textured soil was too limited for any definite conclusion to be made. Legumes grown on the finer textured soils responded to sulphur to a much lesser extent.

Legumes grown in the Cooking Lake district failed to show any response to sulphur fertilization (Figure 1) possibly because of sulphur brought down in the precipitation. Hoff (1953) measured 4.9 lb. of sulphur/acre for the growing season near Edmonton. Considering the proximity of the industrial plants in Edmonton, it is possible that the area received sufficient sulphur to meet the demands of the crop in the years studied. Responses to sulphur in this area were recorded in 1959 and 1960 (Alberta Advisory Fertilizer Committee, 1959, 1960).

Moisture conditions were for the most part poor throughout the course of this investigation. The entire area was generally dry in 1963 with rainfall varying from 0.7 inches to 3.0 inches from May 1 to July 1. The eastern area generally had more rain than the western portion in 1963 (Appendix 1). In 1964, however, the eastern area was extremely dry. The western area had low precipitation in the early part of the season, but by August 1 over 5 inches had been recorded at most sites.

Rainfall no doubt influenced the responses obtained. Responses

were, however, obtained at some sites where rainfall was low while at other sites no response could be observed with high rainfall.

Soil moisture at lower depths generally was low for all soils studied. In the eastern area surface-soil moisture was also very low. Surface moisture was higher in the western area in 1964. The low rainfall and soil moisture conditions experienced in this study probably resulted in an underestimate of the extent of the sulphur deficiency.

Residual effects of the 1963 sulphur applications were observed at sites 8, 29, 46, 56, 69, and 81. In the case of site 8, sulphur response was not observed on the adjoining 1964 plot, probably because of the low rainfall which was 0.4 inches from May 1 to July 1.

Nitrate nitrogen was, with few exceptions, very low in the surface 6 inches of soil. This is the usual observation on soils growing forage crops. Available phosphorus and potassium were extremely variable from site to site (Tables 3 and 4). Available potassium levels were low in the surface 6-inch depth of most soils; on 24 per cent of the sites it was less than 50 lb./acre. Available phosphorus in the 0-6 inch depth varied from 0 to 236 lb./acre.

In 1963 low amounts of available phosphorus were found on non-responsive soils more frequently than on responsive soils as shown by the following table:

| Available Phosphorus pp2m | Responsive | | Non-responsive | |
|---|---|---|---|---|
| | Number | Per Cent | Number | Per Cent |
| 0- 30 | 7 | 28 | 39 | 73 |
| 31- 60 | 6 | 24 | 5 | 9 |
| 61-120 | 7 | 28 | 7 | 13 |
| 120+ | 5 | 20 | 2 | 4 |
| | 25 | | 53 | |

This observation was in agreement with the work of McClung and de Freitas (1959) and suggested the inclusion of an NPK treatment in the plot design. In 1964 NPK and NPKS treatments were included in the plots design as previously described in material and methods.

Sulphur responses were not, however, observed on the NPKS treatment compared to the NPK treatment when there was no response on the accompanying S, check treatment. The observation could be a result of one or both of the following explanations:

1. The NPK remained on the surface of the soil.

2. NPK were being supplied in sufficient amounts to the plant by the soil to allow a response to S.

Because of the drought conditions which prevailed throughout the study and the immobility of phosphorus, the author favors the former suggestion. Nitrogen is not usually a limiting factor in legume production and potassium deficiencies are uncommon in Alberta. Failure of the legume to respond on soils low in available phosphorus occurred to a lesser extent in 1964 as shown in the accompanying table:

| Available Phosphorus pp2m | Responsive | | Non-responsive | |
|---|---|---|---|---|
| | Number | Per Cent | Number | Per Cent |
| 0- 30 | 7 | 43 | 25 | 64 |
| 31- 60 | 4 | 25 | 6 | 15 |
| 61-120 | 2 | 12 | 7 | 18 |
| 120+ | 3 | 20 | 1 | 3 |
| | 16 | | 39 | |

At 6 sites a yield increase was observed on the NPK treatment and not on the S treatment. This suggests that climatic conditions were not a major factor at these sites and the observation that these soils are

non-deficient in sulphur is more conclusive than at many other sites.

The pH of the surface 0-6 inches ranged from 4.9 to 8.0, the majority of the soils being between pH 6.0 and 7.5.

## B. Laboratory Analyses

### (1) Plant Samples

The ethanol-soluble sulphate content of the legume samples is listed in Table 5. The average ethanol-soluble sulphate is 0.45 ± 0.24* per cent for the alfalfa at the 65 sites that failed to respond to the sulphur and 0.13 ± 0.03 per cent for the alfalfa at the 26 sites that responded to the applied sulphur (excluding sample 42). Including sample 42 the value is 0.17 ± 0.14 per cent. The means are significantly different at the 99 per cent level. These values are considerably higher than those reported by Walker (1959) possibly because of different methods of sampling. Walker collected the samples in jute bags and brought them into the laboratory for drying. In the present study, the method of keeping the samples frozen until they were dry would prevent loss of sulphur during drying. Another important reason for the higher values found in the present study was that different portions of the plant were sampled. Walker collected the entire plant while in this study only the terminal portion of the plant was collected. The ethanol-soluble sulphate content of alsike and red clover samples was very low and results were similar to those reported by Walker (1959).

Separation of the responsive and non-responsive soils could be made on the basis of ethanol-soluble sulphate content of the alfalfa in 86 per cent of the cases if a critical level of 0.17 per cent was used. Walker (1959) used a critical level of 0.09 per cent while Hoff (1953) suggested

* Standard deviation.

Table 5. Ethanol-soluble Sulphate of Legume Samples

| Site | Sulphate (%) | Site | Sulphate (%) |
|---|---|---|---|
| I. Non-responsive Sites: | | | |
| 1 | 1.12 | 55 | 0.24 |
| 2a | 0.72 | 58a | 0.39 |
| 2b | 0.35 | 62 | 0.17 |
| 3 | 0.63 | 65* | 0.22 |
| 4 | 0.51 | 66 | 0.24 |
| 5 | 0.53 | 67 | 0.19 |
| 6 | 0.56 | 68 | 0.39 |
| 7a | 0.39 | 70 | 0.10 |
| 7b | 0.29 | 71 | 0.09 |
| 8b | 0.31 | 73 | 0.29 |
| 9 | 0.73 | 75a | 0.23 |
| 14 | 0.46 | 77 | 0.16 |
| 15a | 0.16 | 78 | 0.14 |
| 16 | 0.29 | 80a* | 0.06 |
| 17 | 0.31 | 83a | 0.22 |
| 18a | 0.27 | 84a | 0.27 |
| 18b | 0.53 | 85 | 0.25 |
| 19 | 0.39 | 86 | 0.55 |
| 22 | 0.58 | 87a | 0.93 |
| 23 | 0.84 | 87b | 0.12 |
| 30 | 0.52 | 88a* | 0.19 |
| 32 | 0.29 | 88b* | 0.09 |
| 33b | 0.26 | 90 | 0.73 |
| 35 | 0.74 | 91 | 0.49 |
| 36 | 0.25 | 92 | 0.35 |
| 37b | 0.46 | 93 | 0.48 |
| 38a | 0.83 | 94 | 0.30 |
| 38b | 0.30 | 95 | 0.53 |
| 39b | 0.51 | 96 | 0.74 |
| 40** | 0.14 | 97 | 0.49 |
| 44 | 0.73 | 98 | 0.47 |
| 45a* | 0.35 | 99 | 0.06 |
| 45b* | 0.30 | 101 | 0.14 |
| 48 | 0.62 | 102** | 0.07 |
| 49 | 0.58 | 103 | 0.76 |
| 50** | 0.11 | 104 | 0.69 |
| 52 | 0.92 | 105 | 0.04 |
| 53 | 0.79 | 106 | 0.16 |

* Alsike
** Red clover
All others Alfalfa

Table 5. (Continued)

| Site | Sulphate (%) | Site | Sulphate (%) |
|---|---|---|---|
| II. Responsive Sites: | | | |
| 8a | 0.12 | 46b | 0.05 |
| 10 | 0.02 | 47 | 0.17 |
| 20 | 0.21 | 56a | 0.24 |
| 24 | 0.16 | 57 | 0.28 |
| 25 | 0.12 | 58b | 0.15 |
| 26 | 0.17 | 59** | 0.03 |
| 27 | 0.06 | 61 | 0.13 |
| 28 | 0.06 | 63** | 0.02 |
| 29 | 0.04 | 64* | 0.02 |
| 31 | 0.08 | 69 | 0.21 |
| 37a | 0.31 | 69b | 0.05 |
| 39a* | 0.15 | 76a | 0.13 |
| 41 | 0.07 | 76b | 0.05 |
| 42 | 0.81 | 81a | 0.04 |
| 46a | 0.09 | 82a | 0.15 |
| | | 100 | 0.26 |

* Alsike
** Red clover
All others Alfalfa

the critical level to be 0.15 per cent. To obtain a standard critical level upon which assessment of the sulphur status of the soil could be made would require standardization of the sample collection methods. The yearly variation proved less important than collection method.

Of the 10 non-sulphur-deficient legumes which had ethanol-soluble sulphate contents lower than 0.17 per cent, three samples (15a, 77, and 106) were within the experimental error. Seven samples (15a, 70, 71, 77, 99, 105, and 106) came from plots where the growth and yield of alfalfa was extremely low because of poor growing conditions. Site 87b had high yields for 6 consecutive cuttings. No explanation can be offered for the low ethanol-soluble sulphate in the legumes grown on this site or on site 78 and 101. Of the 7 alfalfa samples from sulphur-deficient sites higher in ethanol-soluble sulphate than 0.17 per cent, 2 (42 and 100) were from crops in which the response to sulphur was on the questionable side. No explanation is offered for the other 5. It should be noted here that the deficient and non-deficient soils were separated without the aid of the ethanol-soluble sulphate data, but rather on visual observation and yield data previously described.

With one exception, the alsike clover could be separated into responsive and non-responsive categories using a critical level of 0.04 per cent as suggested by Walker (1959). The entire above ground portion of the plant was sampled in both Walker's and the present study.

Red clover could be separated into the two categories using a critical level of 0.07 per cent ethanol-soluble sulphate. However, the number of samples was too limited for any definite trends to be established. Walker (1959) could find no difference between red clover grown on sulphur-deficient and non-deficient soil.

The results of the nitrogen analyses are reported in Table 6. The values are higher than those reported by Walker (1959) probably because

Table 6. Total Nitrogen Analyses of Legumes
(1963 Plots)

| Site | Nitrogen (%) Check Plot | Nitrogen (%) S. Plot | Site | Nitrogen (%) Check Plot | Nitrogen (%) S. Plot |
|---|---|---|---|---|---|
| I. Non-responsive Sites: | | | | | |
| 1 | 3.102 | 3.050 | 55 | 4.148 | 4.113 |
| 2 | 3.369 | 3.351 | 58 | 3.839 | 4.052 |
| 3 | 3.593 | 3.953 | 62 | 3.310 | 3.666 |
| 5 | 3.362 | 3.236 | 66 | 3.842 | 3.983 |
| 6 | 3.210 | 3.427 | 67 | 4.025 | 4.307 |
| 7 | 3.957 | 3.892 | 70 | 2.903 | 3.384 |
| 9 | 3.505 | 3.474 | 71 | 3.211 | 3.053 |
| 15 | 3.698 | 3.747 | 72 | 3.238 | 3.823 |
| 16 | 3.433 | 3.359 | 75 | 3.602 | 3.942 |
| 18 | 3.241 | 3.388 | 77 | 3.897 | 4.084 |
| 19 | 3.353 | 3.496 | 78 | 4.279 | 4.057 |
| 22 | 3.701 | 3.934 | 80 | 3.618 | 3.061 |
| 23 | 3.559 | 3.337 | 83 | 4.188 | 4.043 |
| 30 | 3.519 | 3.736 | 84 | 3.713 | 3.736 |
| 35 | 3.973 | 3.089 | 85 | 3.713 | 3.736 |
| 38 | 3.742 | 3.399 | 86 | 3.779 | 3.676 |
| 40 | 3.247 | 3.434 | 87 | 4.436 | 4.173 |
| 44 | 4.256 | 4.243 | 88 | 3.024 | 3.220 |
| 45 | 2.713 | 3.030 | 90 | 3.607 | 3.572 |
| 48 | 3.877 | 3.941 | 103 | 4.098 | 4.268 |
| 50 | 3.455 | 3.413 | 104 | 3.640 | 3.849 |
| 52 | 3.651 | 3.503 | 105 | 2.743 | 3.393 |
| 53 | 3.941 | 4.281 | 106 | 3.846 | 3.920 |
| 54 | 4.291 | 4.375 | | | |
| II. Responsive Sites: | | | | | |
| 8 | 3.554 | 4.103 | 39 | 3.142 | 3.356 |
| 10 | 3.115 | 3.545 | 42 | 3.071 | 3.179 |
| 12* | 3.229 | 3.933 | 46 | 2.720 | 2.891 |
| 20 | 3.716 | 4.013 | 47 | 2.836 | 4.009 |
| 24 | 3.355 | 3.913 | 56 | 3.593 | 3.722 |
| 25 | 3.699 | 3.978 | 57 | 3.896 | 4.208 |
| 26 | 3.801 | 3.825 | 59 | 3.135 | 3.982 |
| 27 | 3.413 | 4.081 | 61 | 2.698 | 4.129 |
| 28 | 3.467 | 3.938 | 64 | 2.726 | 3.842 |
| 29 | 3.285 | 3.976 | 69 | 3.266 | 4.064 |
| 31 | 2.752 | 3.512 | 76 | 3.722 | 4.045 |
| 37 | 3.557 | 3.789 | 81 | 3.599 | 4.199 |
| | | | 82 | 3.851 | 3.935 |

* Response observed in second cutting only.

of the portion of the plant collected and the handling of the samples prior to analyses. The average nitrogen content of the legumes grown on all check plots was 3.53 $\pm$ 0.42 per cent. The sulphur application increased the nitrogen content of the legume grown on sulphur-deficient soil in every case studied, the average increase being 0.52 $\pm$ 0.36 per cent, a significant increase at the 99 per cent level. This represented about a 3.3 per cent increase in the protein content. The average increase in the nitrogen content of legumes, grown on sulphur non-deficient soil fertilized with sulphur, was 0.05 $\pm$ 0.30 per cent. In 22 of the 46 samples the nitrogen content was actually decreased. The increase in this case was not significant. The total nitrogen analysis did not appear to be a diagnostic criterion in separating sulphur-deficient and non-deficient soils.

Some characterization data for the profiles selected for detailed study are given in Table 7. All of the analyses reported subsequently, with the exception of mechanical analyses, are based on air dry soil; however, the hygroscopic water content is listed to permit conversion to the oven-dry basis.

Profiles B, C, D, E, G, and H were classified as Orthic Grey Wooded. Profile A was a Humic Eluviated Gleysol while F was an Orthic Dark Grey. Five profiles (D, E, F, G, and H) were developed on glacial till and the other 3 were developed on lacustrine deposits (A, B, and C). The texture of the profiles ranged from a loamy sand to a clay, the clay content ranging from 2 to 56 per cent.

Carbon to nitrogen ratios ranged from 8.3 to 18.2. Virgin Grey Wooded profiles usually have wide carbon to nitrogen ratios in the L-H horizon and this ratio becomes narrower upon cultivation as the L-H horizon is incorporated into the Ae and upper Bt horizons. The high

Table 7. Mechanical Analyses; pH, Carbon, Nitrogen, and Hygroscopic Moisture of the Selected Profiles

| Sample | Mechanical Analyses % S | % Si | % C | Class | pH | % C | % N | C/N | Hygroscopic Moisture |
|---|---|---|---|---|---|---|---|---|---|
| I. Non-responsive Soils: | | | | | | | | | |
| A1 | 37 | 41 | 22 | L. | 6.8 | 2.70 | 0.222 | 12.2 | 1.86 |
| A2 | 32 | 33 | 35 | C.L. | 6.7 | 0.53 | 0.064 | 8.3 | 2.27 |
| A3 | 36 | 31 | 33 | C.L. | 7.0 | 0.50 | 0.047 | 10.6 | 2.38 |
| A4 | 26 | 34 | 41 | C. | 7.4 | 0.56 | 0.043 | 13.0 | 2.39 |
| B1 | 28 | 53 | 19 | Si.L. | 6.7 | 2.51 | 0.204 | 12.3 | 1.82 |
| B2 | 23 | 49 | 28 | C.L. | 6.7 | 0.99 | 0.089 | 11.1 | 2.02 |
| B3 | 30 | 36 | 34 | C.L. | 7.2 | 0.94 | 0.059 | 15.9 | 2.25 |
| B4 | 34 | 37 | 29 | C.L. | 7.7 | 1.02* | 0.040 | -- | 1.92 |
| C1 | 9 | 45 | 45 | Si.C. | 5.7 | 2.14 | 0.257 | 8.3 | 3.14 |
| C2 | 9 | 35 | 56 | C. | 4.7 | 0.73 | 0.093 | 8.8 | 3.63 |
| C3 | 5 | 48 | 47 | Si.C. | 6.9 | 0.92 | 0.084 | 10.7 | 4.30 |
| C4 | 4 | 32 | 65 | H.C. | 7.6 | 1.59* | 0.070 | -- | 3.86 |
| D1 | 25 | 73 | 2 | Si.L. | 6.9 | 2.18 | 0.163 | 14.4 | 1.19 |
| D2 | 41 | 40 | 18 | L. | 6.5 | 0.48 | 0.050 | 9.6 | 1.21 |
| D3 | 34 | 31 | 35 | C.L. | 6.3 | 0.49 | 0.048 | 10.2 | 2.20 |
| D4 | 34 | 32 | 34 | C.L. | 7.2 | 0.60 | 0.042 | 15.3 | 1.96 |
| II. Responsive Soils: | | | | | | | | | |
| E1 | 26 | 65 | 9 | Si.L. | 7.0 | 2.40 | 0.141 | 18.0 | 1.10 |
| E2 | 38 | 37 | 25 | L. | 6.0 | 0.44 | 0.045 | 9.8 | 1.43 |
| E3 | 37 | 32 | 31 | C.L. | 5.4 | 0.52 | 0.042 | 12.4 | 1.99 |
| E4 | 43 | 34 | 23 | L. | 5.8 | 0.35 | 0.034 | 10.3 | 1.65 |
| F1 | 74 | 20 | 7 | S.L. | 6.4 | 1.60 | 0.112 | 15.3 | 0.84 |
| F2 | 70 | 21 | 9 | S.L. | 6.5 | 0.63 | 0.057 | 11.1 | 0.77 |
| F3 | 70 | 14 | 16 | S.L. | 6.5 | 0.34 | 0.025 | 13.6 | 0.64 |
| F4 | 83 | 9 | 7 | L.S. | 5.4 | 0.39 | 0.038 | 10.3 | 1.46 |
| G1 | 41 | 37 | 22 | L. | 6.3 | 1.36 | 0.111 | 13.3 | 1.45 |
| G2 | 33 | 38 | 39 | C.L. | 5.1 | 0.61 | 0.063 | 9.7 | 2.40 |
| G3 | 17 | 44 | 39 | Si.C.L. | 5.2 | 0.59 | 0.060 | 9.8 | 2.58 |
| G4 | 27 | 35 | 37 | C.L. | 5.2 | 0.56 | 0.049 | 11.4 | 2.29 |
| H1 | 34 | 56 | 11 | Si.L. | 6.4 | 3.42 | 0.188 | 18.2 | 1.27 |
| H2 | 36 | 41 | 23 | L. | 5.5 | 0.71 | 0.062 | 12.5 | 1.40 |
| H3 | 40 | 38 | 32 | C.L. | 5.6 | 0.45 | 0.048 | 10.4 | 1.90 |
| H4 | 40 | 38 | 22 | L. | 5.3 | 0.51 | 0.043 | 12.9 | 2.04 |

* Sample contained $CaCO_3$.

values found in the lower depths of profiles A, B, C, and D may be due to the presence of small amounts of $CaCO_3$ which would increase the ratio. Only on samples B4 and C4 could $CaCO_3$ be detected by a simple acid test.

The pH values of the surface samples ranged from 5.7 to 7.0 and except for sample C1, the range was 6.3 to 7.0. There was no clear difference in the surface samples between responsive and non-responsive soils. The pH values of the lower depths of the non-responsive profiles were higher than those of the corresponding depths of the responsive profiles. This may indicate that sulphur deficiency is greater on those soils which are more strongly leached.

The sulphur fractions which were determined are shown in detail in Table 8 and summarized in Table 9. Total sulphur was very low for all horizons, ranging from 24 to 263 ppm. The smaller values were lower than those reported by Wyatt and Doughty (1928) and Bentley et al. (1955) but are in good agreement with those of Lowe (1965) whose method was the same as the one in this study. It is clear from the total sulphur figures that even in soils which failed to respond to sulphur, the amount of reserve sulphur is low when compared to nitrogen. Barring external addition of sulphur to the soil, crop removal would deplete the sulphur supply to a depth of 3 feet in 100 to 200 years. This estimation is assuming that all the sulphur in the soil would become available to plants.

Carbon-bonded sulphur represented, on the average, 36 per cent of the total sulphur and was low compared to Chernozemic profiles (Lowe, 1965). The carbon-bonded sulphur content of the responsive and non-responsive soils was not significantly different at the 95 per cent level. According to Lowe (1963) this fraction does not include organic sulphate nor the alkyl sulphones. An estimate of organic sulphur can be made by

Table 8. Sulphur Fractions of Selected Profiles

| Sample | Total S ppm | HI-S* ppm | C-S** ppm | HI-S+ C-S as % Total S | Extract-able $SO_4^=$ ppm S | (X) Adsorbed Sulphate ppm S | (Y) Easily Soluble Sulphate ppm S | Total (X)+(Y) ppm S |
|---|---|---|---|---|---|---|---|---|
| I. Non-responsive Soils: | | | | | | | | |
| A1 | 156 | 77 | 70 | 92 | 7 | 4 | 4 | 8 |
| A2 | 93 | 44 | 53 | 104 | 13 | 1 | 5 | 6 |
| A3 | 111 | 57 | 22 | 71 | 10 | 4 | 4 | 8 |
| A4 | 159 | 75 | 28 | 65 | 21 | 6 | 14 | 20 |
| B1 | 126 | 79 | 48 | 100 | 9 | 7 | 4 | 11 |
| B2 | 96 | 53 | 21 | 67 | 10 | 6 | 3 | 9 |
| B3 | 181 | 43 | 16 | 33 | 5 | 6 | 4 | 10 |
| B4 | 259 | 74 | 25 | 38 | 7 | 4 | 6 | 10 |
| C1 | 118 | 64 | 90 | 130 | 7 | 9 | 3 | 12 |
| C2 | 106 | 57 | 18 | 71 | 7 | 6 | 4 | 10 |
| C3 | 122 | 58 | 29 | 72 | 9 | 4 | 7 | 11 |
| C4 | 263 | 60 | 18 | 34 | 12 | 11 | 11 | 22 |
| D1 | 93 | 54 | 48 | 108 | 4 | 4 | 7 | 11 |
| D2 | 24 | 9 | 34 | 179 | 1 | 1 | 5 | 6 |
| D3 | 41 | 14 | 24 | 94 | 3 | 2 | 8 | 10 |
| D4 | 126 | 39 | 15 | 43 | 3 | 1 | 3 | 4 |
| II. Responsive Soils: | | | | | | | | |
| E1 | 137 | 65 | 52 | 85 | 4 | 3 | 2 | 5 |
| E2 | 111 | 24 | 12 | 33 | 5 | 4 | 3 | 7 |
| E3 | 100 | 33 | 14 | 47 | 4 | 9 | 2 | 11 |
| E4 | 52 | 20 | 42 | 119 | 2 | 5 | 2 | 7 |
| F1 | 63 | 39 | 19 | 92 | 4 | 1 | 2 | 3 |
| F2 | 37 | 26 | 12 | 102 | 5 | 3 | 3 | 6 |
| F3 | 63 | 17 | 6 | 37 | 4 | 1 | 1 | 2 |
| F4 | 104 | 31 | 34 | 63 | 1 | 3 | 2 | 5 |
| G1 | 74 | 29 | 50 | 107 | 1 | 2 | 2 | 4 |
| G2 | 82 | 17 | 17 | 41 | 1 | 1 | 3 | 4 |
| G3 | 141 | 23 | 16 | 28 | 2 | 2 | 3 | 5 |
| G4 | 207 | 33 | 21 | 26 | 9 | 3 | 2 | 5 |
| H1 | 148 | 90 | 57 | 99 | 8 | 4 | 4 | 8 |
| H2 | 52 | 30 | 14 | 84 | 2 | 5 | 1 | 6 |
| H3 | 56 | 34 | 45 | 80 | 3 | 3 | 1 | 4 |
| H4 | 40 | 29 | 13 | 105 | 1 | 7 | 3 | 10 |

* HI-reducible sulphur.
** Carbon-bonded sulphur.

Table 9. Summary of the Data in Table 8, Giving Average Concentrations for the Profile of the Various Sulphur Fractions and Averages for the Four Responsive and Four Non-Responsive Soils With Appropriate Standard Deviations

| Profile | Total S ppm | HI-S* ppm | C-S** ppm | HI-S+ C-S as % Total S | Extractable $SO_4^{=}$ ppm S | (X) Adsorbed Sulphate ppm S | (Y) Easily Soluble Sulphate ppm S | Total (X)+(Y) ppm S |
|---|---|---|---|---|---|---|---|---|
| I. Non-responsive Soils: | | | | | | | | |
| A | 129 | 63 | 43 | 82 | 13 | 4 | 7 | 11 |
| B | 166 | 62 | 28 | 54 | 8 | 6 | 4 | 10 |
| C | 152 | 60 | 39 | 65 | 9 | 7 | 6 | 13 |
| D | 71 | 29 | 30 | 83 | 3 | 2 | 6 | 8 |
| II. Responsive Soils: | | | | | | | | |
| E | 100 | 36 | 30 | 66 | 3 | 5 | 2 | 7 |
| F | 67 | 28 | 17 | 67 | 4 | 2 | 2 | 4 |
| G | 126 | 26 | 26 | 41 | 3 | 2 | 2 | 4 |
| H | 74 | 46 | 32 | 95 | 4 | 5 | 2 | 7 |
| Ave. for A-D | 130±41 # | 54±17 | 35±7 | 68 | 8±4 | 5±2 | 6±1 | 11 |
| Ave. for E-H | 92±28 | 34±7 | 27±5 | 63 | 4±5 | 4±2 | 2±0 | 7 |

* HI-reducible sulphur.
** Carbon-bonded sulphur.
\# Standard Deviation.

adding carbon-bonded sulphur to organic sulphate. An estimate of organic sulphate is derived by subtracting inorganic sulphate (adsorbed plus easily-soluble sulphate) from HI-reducible sulphur. Such an estimate reveals that organic sulphur comprises 75 to 95 per cent of the total sulphur in the surface horizon and 25 to 90 per cent of the total sulphur in the lower horizon studied. Total carbon is also low when compared to Chernozemic soils, thus further confirming the data for organic sulphur.

The HI-reducible sulphur content was of the same order of magnitude as that found by Lowe (1965) for Grey Wooded soils, and was also similar to the results of Spencer and Freney (1960) for some Podzolic soils in Australia. This fraction represented on the average 40 per cent of the total sulphur. The average HI-reducible sulphur for the non-responsive and responsive profiles was 54 $\pm$ 17 ppm and 34 $\pm$ 7 ppm, respectively (Table 9), the difference being significant at the 99 per cent level. Profiles D and H do not fit the general trend, profile D having lower values for all depths and profile H having a high value for the surface horizon (Figure 2). Explanations for profiles D and H will be given subsequently.

The phosphate solution (500 ppm P) (Ensminger, 1954) extracted water-soluble sulphate as well as adsorbed sulphate. This fraction has been previously referred to as extractable sulphate. While the amount of sulphur extracted represented only 2 to 10 per cent of the total, significantly more sulphate was extracted from the non-responsive profiles than from the responsive profiles, the respective averages and standard deviations being 8 $\pm$ 4 ppm and 4 $\pm$ 0.5 ppm (Table 9, Figure 2). There is slight overlapping of the ranges of values for non-responsive and responsive sites. McClung and de Freitas et al. (1959) reported that plants are unable to grow when the level of acetate-extractable sulphate

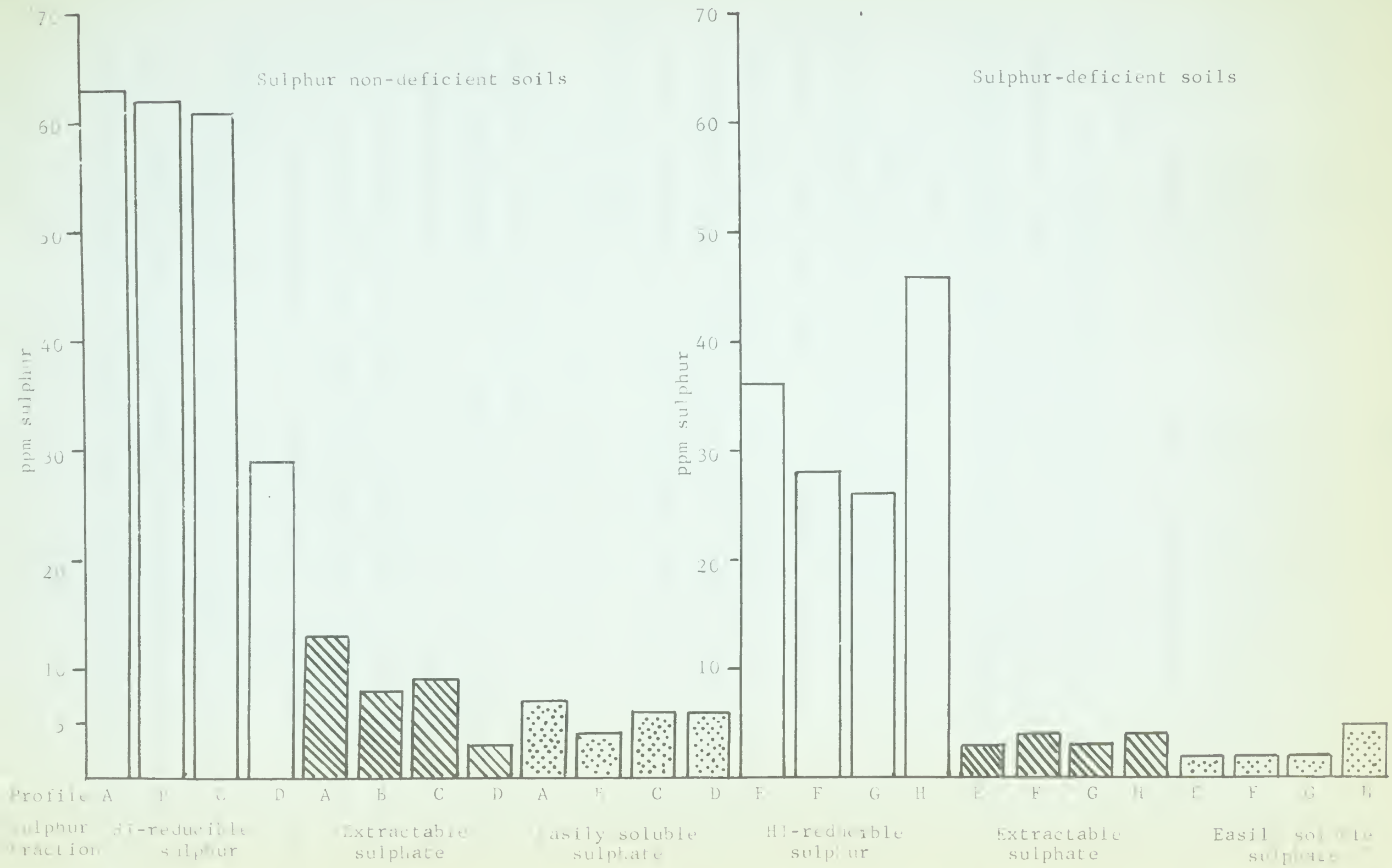



Figure 4. Hi-reducible sulphur, extractable sulphate and easily soluble sulphate of sulphur deficient and non-deficient soils

fell below 2 ppm. In all the sulphur responsive soils studied herein, however, there was growth on the check plot. Using a critical level of 6 ppm the responsive and non-responsive soils, except for profile D, could be separated on the basis of the average extractable sulphate for the profile.

Adsorbed sulphate was low, ranging from a low of 2 ppm for 3 profiles up to 7 ppm. No separation of the profiles was possible using the adsorbed sulphate data, the means being not significantly different at the 95 per cent level.

A significant difference was found between the means of the easily-soluble sulphate content of the responsive and non-responsive profiles (Figure 2). The levels of this fraction, which is essentially water-soluble sulphate, were low in all profiles, the values ranging from 1 to 14 ppm for all the horizons of the 8 profiles studied. Water-soluble sulphate has not been considered to correlate very highly with legume yields (Spencer and Freney, 1960).

The adsorbed plus easily-soluble sulphate values were of the same magnitude as the extractable sulphur content. Based on the higher phosphorus concentration of the solution used to extract adsorbed sulphate and preliminary work by Lowe (1961), it was expected that the adsorbed plus easily-soluble sulphate would be higher than the extractable sulphate values. In 8 samples the values were lower and in 24 the values were higher. Only in samples A2, A3, and G4 did the lower difference appear significant.

All of the sulphur fractions were low in quantity in profile D. Although the soils could be separated into the responsive and non-responsive categories on the bases of 3 sulphur fractions by including profile D,

separation would have been much better excluding it. As pointed out previously, the responsive soils were put into the responsive category with more certainty than the non-responsive soils were put into the non-responsive category. It is suggested that profile D may not be part of the same population as the other non-responsive soils and may actually show sulphur deficiency symptoms in another year. Profile H, a responsive soil, had high concentrations of sulphur in the surface horizon. Using the estimate of organic sulphur previously described, profile H had 97 per cent organic sulphur in the surface 6 inch depth. The C/N ratio was also wide for the sample from this depth indicating that the organic matter may be in a more undecomposed state than in the other profiles and thus the sulphur would be in a form unavailable to plants.

It should be noted that the extreme cases of sulphur-deficient and non-deficient soils were chosen in the Grey Wooded soil zone. In spite of this, only the HI-reducible sulphur, extractable sulphur, and the easily-soluble sulphur permitted separation of the responsive and non-responsive soils. These separations were not completely reliable. In no case could the extent of the sulphur deficiency be estimated. It is the author's opinion that the extractable sulphate and easily-soluble sulphate contents of the soil may be a better diagnostic criterion because the amounts extracted are of the same magnitude as plant uptake. HI-reducible sulphur represents a greater amount of sulphur than is taken up by plants; however, it does represent the more available reserve of sulphur.

It is evident that in the plant-soil system the amount of available sulphur at any time is the result of a number of processes. The most important ones may be the mineralization of organic matter by micro-organisms and the removal of sulphur from the soil by crops. It is also

suggested that atmospheric sulphur may be exerting an external influence on the system so that a clear distinction between sulphur-deficient and non-deficient soils based on soil chemistry is very difficult.

## SUMMARY AND CONCLUSION

The extent of sulphur deficiency in the Grey Wooded soils of Alberta is not fully known. Newton (1936) demonstrated sulphur deficiency at the Breton plots in the early 1930's. Subsequently fertilizer tests have shown many Grey Wooded soils to be sulphur-deficient. Walker (1959) found that not all Grey Wooded soils exhibited sulphur deficiency in an area south and west of Edmonton. It was the purpose of this investigation to study the sulphur status of soils in the Grey Wooded and Dark Grey soil zones in an area generally north of Edmonton (Figure 1).

$Na_2SO_4$ applied at the rate of 20 lb. elemental sulphur/acre increased the yield of legume at 34 of 133 sites, the yield increases being determined on the basis of visual observation and square yard samples. Moisture conditions were below normal in a large percentage of the area in both 1963 and 1964. It is therefore suggested that the above estimation of the extent of sulphur deficiency is an underestimate of the sulphur status of the soils studied.

The nitrate nitrogen content of the soils was generally low. Available phosphorus (0.03N $H_2SO_4$, 0.03 N $NH_4F$ extraction) and available potassium (Spurway method) varied widely from site to site.

The application of NPK did not increase the growth of legumes enough to produce a sulphur deficiency in the soils where no sulphur deficiency had been demonstrated with a straight sulphur treatment.

Sulphur deficiency was not confined to any geographic area nor to any soil series. However, certain soils exhibited sulphur deficiency more frequently than did others.

The ethanol-soluble sulphate content of the legumes grown on sulphur-deficient soils was lower than in legumes grown on sulphur-

sufficient soils. Using a critical level of 0.17 per cent ethanol-soluble sulphate for alfalfa, responsive and non-responsive soils could be separated with 84 per cent reliability.

Sulphur application increased the nitrogen content of the legumes grown on sulphur-deficient soil by an average of 0.52 per cent. No difference was found on the legumes grown on non-deficient soils.

Four distinctly responsive and 4 non-responsive soils were selected for a comprehensive study of sulphur fractions. Four depths were studied in each soil. In these 8 soils texture varied from a loamy sand to a clay. The pH of the horizons varied from 5.1 to 7.7. The lower 2 depths of the responsive soils were more acid than were the corresponding depths of the non-responsive soils.

Carbon to nitrogen ratios ranged from 8.3 to 18.2, the highest values being found in the surface 6 inches of soil. The total sulphur content of the selected soils was low in all horizons studied, ranging from 24 to 263 ppm. The non-responsive profiles had significantly more HI-reducible sulphur, more extractable sulphur, and more easily-soluble sulphate than did the responsive profiles. However, the separation could not be made with 100 per cent reliability. The responsive and non-responsive profiles were not significantly different in carbon-bonded sulphur content nor in adsorbed sulphate content. All the sulphur fractions investigated were low in quantity compared to Chernozemic soils (Lowe, 1965).

It is evident that dynamic processes are in operation within the soil and that the amount of sulphur in any extract is only a static representation of the whole picture. Influences external to the soil, such as the addition of sulphur from the atmosphere, are probably also important.

# BIBLIOGRAPHY

Adams, H. R. 1924. Some effects of sulphur on crops and soil. Soil Sci. 18: 111-115.

Aiyar, S. P. 1945. The significance of sulphur in the manuring of rice. Indian J. Agric. Sci. 15: 283-297.
Seen in Commonwealth Bureau of Soils Bibliography on Sulphur in Plant Nutrition; Sulphur Deficiency. Nov. 1956.

Alberta Advisory Fertilizer Committee. 1959. "Fertilizer tests in Alberta." pp. U21-U47.

Alberta Advisory Fertilizer Committee. 1960. "Fertilizer tests in Alberta." pp. U2-U17.

Alberta Advisory Fertilizer Committee. 1963. "Fertilizers in Alberta" Alberta Department of Agriculture. Pub. 70.

Alway, F. J., A. W. Marsh, and W. J. Methley. 1937. Sufficiency of atmospheric sulphur for maximum crop yields. Soil Sci. Soc. Amer. Proc. 2: 229-238.

Anderson, A. J., and O. Spencer. 1950. Sulphur in metabolism of legumes. Australian J. Sci. Research 3: 431-440.

Anderson, A. J. 1952. The significance of sulphur deficiency in Australian soils. J. Australian Inst. Agr. Sci. 18: 135-139.

Araki, M. 1954. (Sulphur deficiency in wheat). Nagyo. Oyobi. Engei 29: 1447-1448.
Seen in Commonwealth Bureau of Soils Bibliography on Sulphur in Plant Nutrition; Sulphur Deficiency. Nov. 1956.

Asghar, A. G., M. A. Qayyum, and G. M. Rana. 1957. A method for volumetric estimation of sulphates in soils and irrigation water. Soil Sci. 83: 239-241.

Bardsley, C. E. Jr., and Howard V. Jordan. 1957. Sulphur availability in seven southeastern soils as measured by growth and composition of white clover. Agron. J. 49: 310-312.

Bardsley, C. E., and J. D. Lancaster. 1960. Determination of reserve sulphur and soluble sulphates in soils. Soil Sci. Soc. Amer. Proc. 24: 265-268.

Bardsley, C. E., and V. J. Kilmer. 1963. Sulphur supply of soils and crop yields in the southeastern United States. Soil Sci. Soc. Amer. Proc. 27: 197-199.

Barrow, N. J. 1961. Studies on mineralization of sulphur from soil organic matter. Aust. J. Agr. Research 12: 306-319.

Bentley, C. F., D. J. Hoff, and D. B. Scott. 1955. Fertilizer studies with radioactive sulphur. II. Can. J. Agr. Sci. 35: 264-281.

Bertolacini, R. J., and J. E. Barney. 1957. Colorimetric determination of sulphate with barium chloranilate. Anal. Chem. 29: 281-283.

Cairns, R. R., and A. C. Richer. 1960. Comparitive study of a sulphur responsive and a non-responsive grey wooded soil. Can. J. Soil Sci. 40: 246-254.

Cassidy, N. G. 1956. An electrometric method for determining sulphate ion. Analyst. 81: 169-175.

Chao, T. T., M. E. Harward, and S. C. Fang. 1962a. Soil constituents and properties in the adsorption of sulphate ions. Soil Sci. 94: 276-283.

Chao, T. T., M. E. Harward, and S. C. Fang. 1962b. Movement of $S^{35}$ tagged sulphate through soil columns. Soil Sci. Soc. Amer. Proc. 26: 27-32.

Chao, T. T., M. E. Harward, and S. C. Fang. 1962c. Adsorption and desorption phenomena of sulphate ions in soils. Soil Sci. Soc. Amer. Proc. 26: 234-237.

Chao, T. T., M. E. Harward, and S. C. Fang. 1963. Cationic effects on sulphate adsorption by soils. Soil Sci. Soc. Amer. Proc. 27: 35-38.

Chesnin, L., and C. H. Yien. 1950. Turbidimetric determination of available sulphates. Soil Sci. Soc. Amer. Proc. 15: 149-151.

Conrad, J. P., H. L. Hall, and B. A. Charigale. 1947. Sulphur fertilization of legumes in the Upper Ojai Valley, California, and the resulting effects on the following nonlegumes. Soil Sci. Soc. Amer. Proc. 12: 275-277.

Conrad, J. P. 1954. Sulphur fertilization in California and some related factors. Soil Sci. 70: 43-54.

Cormack, D. V., C. F. Bentley, and D. B. Scott. 1951. Fertilizer studies with radioactive sulphur. I. Sci. Agr. 31: 41-51.

Cressman, H. K., and J. F. Davis. 1961. Little sulphur is lost from the soil. Crops and Soils 14: 22.

Cressman, H. K., and S. F. Davis. 1962. Sources of sulphur for crops in Michigan and effect of sulphur fertilization on plant growth and composition. Agron. J. 54: 341-344.

Crocker, W. C. 1945. Sulphur deficiency in soils. Soil Sci. 60: 149-155.

DeLong, W. A., and L. E. Lowe. 1962. Note on carbon-bonded sulphur in soil. Can. J. Soil Sci. 42: 223.

Dept. of Soil Science, University of Saskatchewan. 1955. University of Saskatchewan Agr. Extension Bull. 122.

Dijshoorn, W., J. E. M. Lampe, and P. F. J. van Burg. 1960. A method of diagnosing the sulphur nutrition status of herbage. Plant and Soil 13: 227-241.

Donald, C. M., and C. H. Williams. 1954. Fertility and productivity of a podzolic soil as influenced by subterranean clover (Trifolium subterranean l.) and superphosphate. Australian Agr. J. Research 5: 664-687.

Doughty, J. L. 1942. The advantages of a soil paste for routine pH determination. Sci. Agr. 22: 135-138.

Dutt, A. K. 1962. Sulphur deficiency in sugar cane. Empire J. Exptl. Agr. 30: 257-262.

Ensminger, L. E. 1954. Some factors affecting the adsorption of sulphate by Alabama soils. Soil Sci. Soc. Amer. Proc. 18: 259-264.

Ensminger, L. E. 1958. Sulphur in relation to soil fertility. Agricultural Exp. Sta. of the Alabama Polytechnic Institute Bull. 312.

Ergle, D. R., and F. M. Eaton. 1951. Sulphur nutrition of cotton. Plant Physiol. 26: 639-654.

Ergle, D. R. 1953. Effect of low nitrogen and sulphur supply on their accumulation in the cotton plant. Botan. Gaz. 114: 417-426.

Evans, C. A., and C. D. Rost. 1945. Total organic sulphur and humus sulphur of Minnesota soils. Soil Sci. 59: 125-137.

Fox, R. L., H. M. Atesalp, D. H. Kampbell, and H. F. Rhoades. 1964. Factors influencing the availability of sulphur fertilizers to alfalfa and corn. Soil Sci. Soc. Amer. Proc. 28: 406-408.

Fox, R. L., R. A. Olson, and H. F. Rhoades. 1964. Evaluating the sulphur status of soil by plant and soil tests. Soil Sci. Soc. Amer. Proc. 28: 243-246.

Frederick, L. R., R. L. Starkey, and W. Segal. 1957. Decomposability of some organic sulphur compounds in soil. Soil Sci. Soc. Amer. Proc. 21: 287-292.

Freney, J. R. 1958. Determination of water-soluble sulphate in soils. Soil Sci. 86: 241-244.

Freney, J. R. 1961. Some observations on the nature of organic sulphur compounds in soil. Australian J. Agr. Research. 12: 424-432.

Freney, J. R., N. S. Barrow, and K. Spencer. 1962. A review of certain aspects of sulphur as a soil constituent and plant nutrient. Plant and Soil 17: 295-308.

Goldschmit, W. B. 1951. The total sulphur content of some cultivated low yield soils and its probable relation to profitable crop production. S. Afric. J. Sci. 47: 261-262.

Greenwood, M. 1955. Sulphur deficiency in groundnuts in Northern Nigeria. Trans. Intern. Congr. Soil Sci. 3: 245-251.

Gunnarsson, O. 1959. Notes on sulphur and sulphur deficiency. Växtnär. nytt. 15: No. 5. 20-28.
Seen in Commonwealth Bureau of Soils Bibliography on Sulphur Deficiency. 1963.

Hart, M. G. R. 1962. Observations on the source of acid empoldered mangrove soils; formation of elemental sulphur. Plant and Soils 17: 87-98.

Harward, M. E., T. T. Chao, and S. C. Fang. 1962a. Soil properties and constituents in relation to mechanisms of sulphate adsorption. In Radioisotopes in Soil-Plant Nutrition Studies pp. 93-114. International Atomic Energy Agency, Vienna.

Harward, M. E., T. T. Chao, and S. C. Fang. 1962b. The sulphur status and sulphur supplying power of Oregon soils. Agron. J. 54: 101-106.

Hesse, P. R. 1957. Sulphur and nitrogen changes in fareast soils in East Africa. Plant and Soil 9: 86-96.

Hilder, E. J. 1954. Some aspects of sulphur as a nutrient for pastures in New England soils. Australian J. Agric. Research 5: 39-54.

Hoff, D. J. 1953. Fertility studies with radioactive and non-radioactive sulphur on some grey wooded soils of Alberta. M. Sc. Thesis, Univ. of Alberta.

Illinois Agricultural Experimental Station. 1937. Illinois Agr. Expt. Sta. 49th Ann. Rpt. 14-17.

Jackson, M. L. 1963. Aluminum bonding in soils. A unifying principle in soil science. Soil Sci. Soc. Amer. Proc. 27: 1-10.

Jensen, M. C., C. G. Lewis, and G. O. Baker. 1951. Idaho Agr. Exp. Sta. Research Bull. 19.

Johnson, C. M., and H. Nishita. 1952. Microestimation of sulphur in plant materials, soils and irrigation waters. Anal. Chem. 24: 736-742.

Johnson, C. M., H. Nishita, and T. H. Arkley. 1954. Microestimation of sulphur in plant materials, soils and irrigation waters. Anal. Chem. 26: 525.

Jones, M. B. 1962. Total sulphur and sulphate sulphur content in subterranean clover as related to sulphur responses. Soil Sci. Soc. Amer. Proc. 26: 482-484.

Jones, M. B. 1963. Effect of sulphur applied and date of harvest on yield, sulphate sulphur concentration and total sulphur uptake of five annual grassland species. Agron. J. 55: 251-255.

Jones, M. B., and W. E. Martin. 1964. Sulphate-sulphur concentration as an indicator of sulphur status in various California dryland pasture species. Soil Sci. Soc. Amer. Proc. 28: 539-541.

Jordan, H. V., and L. E. Ensminger. 1958. The role of sulphur in soil fertility. Advances in Agron. 10: 407-434.

Junge, C. E., and R. T. Wirtby. 1958. The concentration of chloride, sodium, potassium, calcium and sulphate in rainwater over the United States. J. Meteorol. 15: 417-425.

Kilmer, V. J., and D. C. Nearpass. 1960. The determination of available sulphur in soils. Soil Sci. Soc. Amer. Proc. 24: 337-340.

Kitson, R. E., and M. G. Mellon. 1944. Colorimetric determination of phosphorus as molybidivanadophosphoric acid. Ind. Eng. Chem. Anal. 16: 379-383.

Laboratory Equipment Corporation. 1957. Instruction manual for operation of Leco sulphur determinators.

Laboratory Equipment Corporation. 1959. Instruction manual for operation of Leco carbon analyzer.

Lawton, K. 1955. The chemical composition of soils. A.C.S. Monograph No. 126. Chemistry of the Soil. Ed. by F. E. Bear. pp. 53-84. Reinhold Pub. Corp., New York.

Lipman, J. G., and A. B. Conybeare. 1936. Preliminary note on the inventory and balance sheet of plant nutrients in the United States. New Jersey Agr. Expt. Sta. Bull. 607.

Little, R. C. 1958. Sulphur in Soils. III. A study of the readily soluble sulphate content and of the total sulphur content of soil. J. Sci. Food Agric. 5: 273-281.

Lowe, L. E., and W. A. DeLong. 1961. Aspects of the sulphur status of three Quebec soils. Can. J. Soil Sci. 41: 141-146.

Lowe, L. E. 1963. The sulphur status of Quebec soils with particular reference to the amount of organically combined sulphur and the nature of its combination. Ph. D. Thesis, McGill University, Montreal, Quebec.

Lowe, L. E., and W. A. DeLong. 1963. Carbon bonded sulphur in selected Quebec soils. Can. J. Soil Sci. 43: 151-155.

Lowe, L. E. 1964. An approach to the study of the sulphur status of soils and its application to selected Quebec soils. Can. J. Soil Sci. 44: 176-179.

Lowe, L. E. 1965. Sulphur fractions of selected Alberta soil profiles: I. Soils of the Chernozemic and Podzolic orders. Unpublished, submitted to Can. J. Soil Sci.

Lutz, J. A. Jr. 1957. Sulphur in precipitation and atmosphere of Virginia and crop response to sulphur fertilization. Virginia Agr. Exp. Sta. Bull. 482.

Madanov, P. C. 1946. Nitrogen-sulphur ratio in the humus of steppe-soils. Pedology. 517-528.

Martin, W. E. 1958. Sulphur deficiency widespread. Calif. Agr. 12. No. 11: 10-13.

Mattson, S. 1931. The laws of soil colloidal behavior: VI. Amphoteric behavior. Soil Sci. 32: 343-365.

McClung, A. C., L. M. M. de Freitas, and W. L. Lott. 1959. Analyses of several Brazilian soils in relation to plant responses to sulphur. Soil Sci. Soc. Amer. Proc. 23: 221-224.

McClung, A. C., and L. M. M. de Freitas. 1959. Sulphur deficiency in soils from Brazilian campos. Ecology 40: 315-317.

McClung, A. C., and L. R. Quinn. 1959. Sulphur and phosphorus responses of grass. I.B.E.C. Research Institute Bull. 18.

McKill, C. M., and W. A. Williams. 1960. A lysimeter study of sulphur fertilization of an annual range soil. J. Range Management 13: 113-117.

McLachlan, K. D. 1955. Phosphorus, sulphur and molybdenum deficiencies in soils from eastern Australia in relation to nutrient supply and some characteristics of soil and climate. Australian J. Agr. Research 6: 673-684.

Meeker, E. W., and W. C. Wagner. 1933. Titration of ammonia in presence of boric acid. Ind. Eng. Chem. Anal. Ed. 5: 396-398.

Mehring, A. L., and G. A. Bennett. 1950. Sulphur in fertilizers, manures and soil amendments. Soil Sci. 70: 73-81.

Miller, R. B. 1958. The chemical composition of rainwater at Taita, New Zealand. New Zealand J. Sci. 4: 844-853.

Neller, J. R. 1959. Extractable sulphate-sulphur in soils of Florida in relation to amount of clay in the profile. Soil Sci. Soc. Amer. Proc. 23: 346-348.

Newton, J. D. 1936. The fertilizing value of sulphate in natural "alkali" for grey wooded soils. Sci. Agric. 16: 241-243.

Pratt, P. F., and F. L. Blair. 1962. Buffer methods for estimating lime and sulphur applications for pH control of soils. Soil Sci. 93: 329-332.

Putnam, H. D., and E. L. Schmidt. 1959. Studies on the free amino-acid fraction of soils. Soil Sci. 87: 22-27.

Renner, Ruth, C. F. Bentley, and L. W. McElroy. 1963. Nine essential amino acids in the protein of wheat and barley grown on sulphur-deficient soil. Soil Sci. Soc. Amer. Proc. 17: 270-273.

Rossiter, R. C. 1952. Studies on the nutrition of pasture plants in the southwest of Western Australia. III. The effect of sulphur on the growth of subterranean clover (Trifolium subterranean L.) Aust. J. Agric. Research 3: 7-15.

Sanford, J. O., and J. D. Lancaster. 1962. Biological and chemical evaluation of the readily available sulphur status of Mississippi soils. Soil Sci. Soc. Amer. Proc. 26: 63-65.

Schell, W. R., and J. V. Jordan. 1959. Anion exchange studies of pure clays. Plant and Soil 10: 303-318.

Soofi, G. S., and H. D. Fuehring. 1964. Nutrition of corn on a calcareous soil: I. Interrelationships of N, P, K, Mg, and S on the growth and composition. Soil Sci. Soc. Amer. Proc. 28: 76-79.

Spencer, K. 1959. Growth and chemical composition of white clover as affected by sulphur supply. Australian J. Agr. Research 10: 500-509.

Spencer, K., and J. R. Freney. 1960. A comparison of several procedures for estimating the sulphur status of soils. Australian J. Agr. Research 11: 948-959.

Spurway, C. H., and K. Lawton. 1949. Soil testing, a practical system of soil fertility diagnosis. Mich. State Coll. Bull. 32.

Starkey, R. L. 1950. Relations of micro-organisms to transformations of sulphur in soils. Soil Sci. 70: 55-65.

Starkey, R. L., W. Segal, and R. A. Manaker. 1953. Sulphur products of the decomposition of methionine and cystine by micro-organisms. Proc. Sixth Int. Congr. Microbiol. Rome 1: 256-257.

Stephens, C. G., and C. M. Donald. 1958. Australian soils and their responses to fertilizers. Advances in Agron. 10: 167-256.

Stauffer, R. S., and R. H. Rust. 1954. Leaching losses, runoff, and percolate from eight Illinois soils. Agron. J. 46: 207-211.

Thomas, M. P., R. H. Hendricks, L. C. Bryner, and G. R. Hill. 1944. A study of the sulphur metabolism of wheat, barley, and corn using radioactive sulphur. Plant Physiol. 19: 227-244.

Thomas, M. P., R. H. Hendricks, and G. R. Hill. 1950a. Sulphur content of vegetation. Soil Sci. 70: 9-19.

Thomas, M. P., R. H. Hendricks, and G. R. Hill. 1950b. Sulphur metabolism in alfalfa. Soil Sci. 70: 19-26.

Thorne, D. W., and H. B. Peterson. 1954. Irrigated soils. The Blakiston Co. Inc., Toronto.

Toogood, J. A., and T. W. Peters. 1953. Comparison of methods of mechanical analysis of soils. Can. J. Agr. Sci. 33: 159-171.

United States Department of Agriculture. 1954. Diagnosis and improvement of saline-alkali soils. L. A. Richards ed. Agr. Handb. 60: U.S.D.A., Washington, D.C.

Waksman, S. A., and R. L. Starkey. 1947. The soil and the microbe. John Wiley and Sons, New York 1, N.Y.

Walker, D. R. 1959. The determination of sulphur deficiency in soils. M.Sc. Thesis, Univ. of Alberta.

Walker, D. R., and C. F. Bentley. 1961. Sulphur fractions of legumes as indicators of sulphur deficiency. Can. J. Soil Sci. 41: 164-168.

Walker, T. W., and A. F. R. Adams. 1958. Studies on soil organic matter. I. Influences of phosphorus content of parent materials on accumulations of carbon, nitrogen, sulphur, and organic phosphorus in grassland soils. Soil Sci. 85: 307-318.

Walker, T. W. 1955. Sulphur responses on pasture in Australia and New Zealand. Soils and Fertilizers 27: 1-8.

Whitehead, P. C. 1964. Soil and plant nutrition aspects of the sulphur cycle. Soils and Fertilizers 27: 1-8.

Williams, C. H., and C. M. Donald. 1957. Changes in organic matter and pH in a podzolic soil as influenced by subterranean clover and superphosphate. Australian J. Agr. Research 8: 179-189.

Williams, C. H., and A. Steinbergs. 1958. Sulphur and phosphorus in some eastern Australian soils. Australian J. Agr. Research 9: 483-491.

Williams, C. H., and A. Steinbergs. 1959. Soil sulphur fractions as chemical indices of available sulphur in some Australian soils. Australian J. Agr. Research 10: 340-352.

Williams, C. H., and A. Steinbergs. 1962. The evaluation of plant available sulphur in soils; chemical nature of sulphate in some Australian soils. Plant and Soil 17: 279-294.

Wyatt, F. A., and J. L. Doughty. 1928. Sulphur in Alberta soils. Sci. Agr. 8: 549-555.

Appendix I. Soil Moisture, 1/3 Atmosphere Percentage, and Rainfall for 1963 Sites

| Site | Soil Moisture in May for Depth in Inches (%) | | | | | 1/3 Atmosphere Percentage for Depth in Inches (%) | | | | | Rainfall (inches) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | |
| 1 | 36 | 16 | 19 | 4 | 16 | 41 | 27 | 33 | 28 | 33 | 1.0 |
| 2 | 36 | 34 | 28 | 21 | 14 | 40 | 43 | 44 | 37 | 26 | 0.7 |
| 3 | 27 | 28 | 20 | 15 | 13 | 36 | 34 | 31 | 25 | 23 | -- |
| 5 | 44 | 37 | 34 | 34 | 35 | 46 | 51 | 50 | 48 | 48 | 0.4 |
| 6 | 41 | 39 | 36 | 29 | 28 | 46 | 50 | 50 | 40 | 42 | -- |
| 7 | 25 | 18 | 15 | 12 | 12 | 24 | 21 | 24 | 25 | 24 | 0.8 |
| 8 | 11 | 14 | 15 | 12 | 13 | 19 | 20 | 19 | 21 | 19 | 2.4 |
| 9 | 38 | 23 | 17 | 16 | 15 | 49 | 27 | 21 | 21 | 19 | 2.0 |
| 10 | 11 | 12 | 12 | 14 | 18 | 26 | 19 | 20 | 21 | 22 | 2.3 |
| 11 | 11 | 10 | 13 | 16 | 16 | 37 | 9 | 14 | 18 | 18 | -- |
| 12 | 10 | 12 | 11 | 10 | 10 | 21 | 19 | 18 | 16 | 17 | -- |
| 13 | 28 | 18 | 13 | 12 | 11 | 26 | 24 | 18 | 19 | 18 | -- |
| 14 | 17 | 10 | 10 | 8 | -- | 21 | 14 | 18 | 15 | 16 | 3.0 |
| 16 | 18 | 13 | 14 | 17 | 16 | 27 | 14 | 17 | 18 | 16 | 3.0 |
| 18 | 17 | 12 | 14 | 16 | 15 | 30 | 15 | 18 | 22 | 23 | 2.3 |
| 19 | 16 | 18 | 17 | 15 | 15 | 28 | 21 | 23 | 23 | 24 | 2.9 |
| 20 | 18 | 18 | 18 | 17 | 15 | 25 | 21 | 21 | 25 | 25 | 1.3 |
| 21 | 26 | 16 | 18 | 15 | 14 | 32 | 19 | 24 | 49 | 46 | -- |
| 22 | 36 | 28 | 23 | 21 | 20 | 29 | 35 | 39 | 36 | 32 | 2.2 |
| 23 | 23 | 24 | 21 | 22 | 26 | 30 | 33 | 27 | 30 | 33 | -- |
| 24 | 14 | 11 | 16 | 8 | -- | 23 | 21 | 14 | 15 | -- | 0.6 |
| 25 | 16 | 12 | 12 | 13 | 14 | 28 | 13 | 15 | 14 | 13 | -- |
| 26 | 15 | 16 | 16 | 15 | 14 | 19 | 34 | 22 | 20 | 23 | -- |
| 27 | 12 | 10 | 11 | 10 | 16 | 12 | 13 | 14 | 15 | 19 | -- |

Appendix I. (Continued)

| Site | Soil Moisture in May for Depth in Inches (%) | | | | | 1/3 Atmosphere Percentage for Depth in Inches (%) | | | | | Rainfall (inches) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | |
| 28 | 13 | 9 | 11 | 18 | 21 | 9 | 6 | 28 | 28 | 26 | 1.3 |
| 29 | 13 | 14 | 14 | 15 | 10 | 14 | 14 | 20 | 22 | 16 | 1.0 |
| 30 | 14 | 12 | 8 | 7 | 6 | 12 | 10 | 9 | 7 | 6 | -- |
| 31 | 23 | 16 | 16 | 15 | 13 | 24 | 16 | 19 | 21 | 20 | -- |
| 33 | 24 | 17 | 15 | 16 | 16 | 28 | 19 | 18 | 20 | 18 | -- |
| 35 | 18 | 13 | 14 | 11 | 14 | 25 | 22 | 27 | 27 | 26 | 2.8 |
| 37 | 13 | 9 | 8 | 14 | 15 | 13 | 7 | 7 | 18 | 20 | 2.5 |
| 38 | -- | 24 | 21 | 19 | 21 | 40 | 23 | 29 | 29 | 29 | 2.9 |
| 39 | 24 | 19 | 22 | 21 | 20 | 27 | 22 | 27 | 25 | 28 | 2.8 |
| 40 | 48 | 16 | 21 | 20 | 23 | 30 | 13 | 29 | 28 | 34 | -- |
| 42 | 57 | 20 | 18 | 19 | 20 | 43 | 25 | 24 | 25 | 25 | -- |
| 44 | 22 | 16 | 17 | 18 | 19 | 27 | 22 | 26 | 30 | 27 | 1.7 |
| 45 | 25 | 24 | 23 | 26 | 30 | 26 | 29 | 33 | 38 | 42 | 1.1 |
| 46 | 21 | 22 | 22 | 21 | 20 | 24 | 24 | 32 | 35 | 29 | 0.5 |
| 47 | 19 | 15 | 11 | 12 | 12 | 22 | 20 | 13 | 16 | 19 | 1.0 |
| 48 | 27 | 23 | 26 | 19 | 16 | 28 | 27 | 31 | 20 | 15 | -- |
| 50 | 29 | 13 | 18 | 18 | 19 | 26 | 21 | 25 | 27 | 28 | 0.8 |
| 52 | 55 | 20 | 19 | 18 | 15 | 27 | 30 | 28 | 27 | 31 | 0.7 |
| 53 | 32 | 20 | 18 | 18 | 19 | 32 | 24 | 23 | 23 | 24 | 0.6 |
| 54 | 18 | 21 | 17 | 17 | 18 | 23 | 27 | 25 | 25 | 24 | -- |
| 55 | 18 | 15 | 13 | 12 | 11 | 18 | 22 | 22 | 23 | 27 | -- |
| 56 | 20 | 17 | 17 | 13 | 15 | 25 | 22 | 24 | 25 | 29 | 0.7 |
| 57 | 19 | 20 | 17 | 14 | 15 | 20 | 24 | 23 | 27 | 28 | 0.9 |
| 58 | 19 | 16 | 19 | 17 | 17 | 27 | 23 | 30 | 32 | 32 | 0.6 |
| 59 | 18 | 17 | 17 | 21 | 20 | 21 | 19 | 24 | 33 | 33 | 0.3 |
| 61 | 15 | 22 | 16 | 30 | 17 | 23 | 17 | 23 | 28 | 35 | 1.5 |
| 62 | 19 | 18 | 15 | 14 | 14 | 25 | 24 | 26 | 25 | 25 | 1.4 |
| 64 | 26 | 21 | 25 | 25 | 27 | 30 | 28 | 34 | 36 | 36 | 1.3 |

Appendix I. (Continued)

| Site | Soil Moisture in May for Depth in Inches (%) | | | | | 1/3 Atmosphere Percentage for Depth in Inches (%) | | | | | Rainfall (inches) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | |
| 66 | 20 | 22 | 22 | 7 | 18 | 34 | 39 | 29 | 26 | 27 | 2.0 |
| 67 | 16 | 18 | 13 | 12 | 15 | 27 | 33 | 51 | 47 | 43 | 1.2 |
| 69 | 13 | 12 | 13 | 15 | 17 | 31 | 7 | 7 | 14 | 22 | 1.5 |
| 70 | 10 | 10 | 16 | 15 | 14 | 14 | 10 | 22 | 18 | 25 | 1.6 |
| 71 | 16 | 16 | 15 | 15 | 15 | 24 | 24 | 25 | 26 | 27 | -- |
| 72 | 16 | 16 | 13 | 15 | 20 | 28 | 21 | 18 | 24 | 29 | -- |
| 73 | 20 | 22 | 18 | 15 | 15 | 26 | 23 | 30 | 29 | 30 | 1.0 |
| 75 | 17 | 17 | 14 | 14 | 15 | 37 | 25 | 24 | 27 | 27 | 1.1 |
| 76 | 14 | 14 | 14 | 10 | 12 | 30 | 23 | 24 | 22 | 23 | 1.0 |
| 77 | 14 | 20 | 15 | 18 | 18 | 30 | 29 | 30 | 32 | 32 | 1.0 |
| 78 | 25 | 30 | 28 | 19 | 16 | 33 | 38 | 37 | 32 | 30 | 2.3 |
| 80 | 28 | 30 | 36 | 34 | 34 | 35 | 37 | 48 | 47 | 50 | 0.8 |
| 81 | 24 | 25 | 19 | 18 | 22 | 23 | 25 | 34 | 29 | 22 | 1.2 |
| 82 | 16 | 25 | 25 | 22 | 14 | 24 | 36 | 39 | 39 | 32 | 1.3 |
| 83 | 26 | 19 | 21 | 20 | 19 | 38 | 33 | 40 | 31 | 31 | 1.3 |
| 84 | 10 | 11 | 16 | 14 | 14 | 28 | 20 | 26 | 23 | 22 | 1.1 |
| 85 | 32 | 33 | 18 | 18 | 17 | 39 | 28 | 24 | 21 | 22 | -- |
| 86 | 17 | 15 | 14 | 18 | 20 | 26 | 28 | 21 | 30 | 30 | 0.6 |
| 87 | 22 | 21 | 9 | 9 | 36 | 28 | 28 | 31 | 34 | 37 | 0.6 |
| 88 | 16 | 21 | 25 | 25 | 24 | 25 | 30 | 26 | 34 | 36 | 0.8 |
| 89 | 18 | 16 | 12 | 13 | 14 | 25 | 30 | 36 | 21 | 40 | -- |
| 90 | 14 | 14 | 10 | 13 | 11 | 24 | 25 | 30 | 44 | 37 | -- |
| 103 | 15 | 15 | 17 | 17 | 19 | 21 | 33 | 35 | 25 | 28 | -- |
| 104 | 29 | 19 | 16 | 16 | 16 | 36 | 27 | 25 | 25 | 25 | 0.8 |
| 105 | 21 | 17 | 15 | 15 | 14 | 27 | 23 | 22 | 21 | 20 | 1.1 |
| 106 | 24 | 24 | 26 | 23 | 20 | 27 | 29 | 34 | 35 | 34 | 0.9 |
| 107 | 19 | 20 | 13 | 14 | 16 | 34 | 30 | 30 | 33 | 36 | 0.6 |

Appendix II. Soil Moisture and Rainfall for 1964 Sites

| Site | Soil Moisture in May for Depth in Inches (%) 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | Rainfall (inches) May 1 - July 1 | Rainfall (inches) July 1 - August 15 |
|---|---|---|---|---|---|---|---|
| 2 | 24 | 24 | 20 | 14 | 13 | 1.6 | 0.9 |
| 4 | 30 | 26 | 20 | 15 | 13 | 1.0+ | 3.2 |
| 7 | 21 | 19 | 14 | 10 | 10 | 1.3 | -- |
| 8 | 18 | 16 | 15 | 11 | 12 | 0.4 | 2.0+ |
| 11 | 13 | 11 | 10 | 11 | 12 | -- | 5.2 |
| 12 | 13 | 13 | 9 | 8 | -- | -- | -- |
| 14 | 29 | 17 | 8 | 12 | 11 | 1.2 | 5.2 |
| 15 | 17 | 13 | 10 | 7 | -- | -- | -- |
| 17 | 19 | 16 | 14 | 11 | 10 | 1.4 | -- |
| 18 | 17 | 12 | 9 | 8 | -- | -- | -- |
| 32 | 19 | 14 | 12 | 10 | 8 | 0.0 | -- |
| 33 | 22 | 17 | 13 | 14 | 15 | 0.4 | 3.7 |
| 34 | 12 | 12 | 10 | 10 | 10 | 0.5 | 2.1 |
| 36 | 14 | 15 | 13 | 13 | 14 | 1.6 | 6.0+ |
| 37 | 11 | 8 | 9 | 11 | 12 | -- | -- |
| 38 | 15 | 13 | 14 | 12 | 13 | -- | -- |
| 39 | 15 | 19 | 19 | 16 | 16 | -- | -- |
| 41 | 15 | 11 | 13 | 15 | 17 | 0.8 | 8.0 |
| 43 | 29 | 17 | 13 | 13 | 21 | 1.0 | 6.0 |
| 45 | 29 | 23 | 18 | 20 | 17 | 2.0 | -- |
| 46 | 20 | 19 | 19 | 14 | 15 | 2.2 | 5.0 |
| 49 | 33 | 22 | 23 | 19 | 20 | 1.0 | 5.2 |
| 51 | 21 | 15 | 15 | 14 | 17 | 1.8 | 5.8 |
| 56 | 31 | 22 | 20 | 16 | 19 | 3.0 | 7.5 |
| 58 | 24 | 20 | 17 | 13 | 13 | 3.9 | 3.0 |
| 60 | 50 | 23 | 20 | 25 | 26 | 3.4 | -- |
| 61 | 21 | 15 | 16 | 16 | 14 | 2.8 | 8.7 |
| 63 | 23 | 20 | 16 | 13 | 13 | -- | -- |

Appendix II. (Continued)

| Site | Soil Moisture in May for Depth in Inches (%) 0-6 | 6-12 | 12-24 | 24-36 | 36-48 | Rainfall (inches) May 1 - July 1 | Rainfall (inches) July 1 - August 15 |
|---|---|---|---|---|---|---|---|
| 65 | 25 | 25 | 29 | 25 | 21 | 3.8 | 5.8 |
| 66 | 26 | 22 | 15 | 13 | 13 | 5.8+ | -- |
| 69 | 14 | 13 | 10 | 11 | 13 | -- | 6.0+ |
| 74 | 22 | 19 | 14 | 12 | 12 | 3.6 | 5.8+ |
| 75 | 26 | 18 | 14 | 14 | 14 | 2.6 | 5.2 |
| 76 | 12 | 10 | 14 | 9 | 9 | 3.1 | 6.0+ |
| 79 | 19 | 20 | 16 | 15 | 19 | 1.8 | -- |
| 80 | 25 | 25 | 27 | 21 | 21 | -- | -- |
| 81 | 20 | 14 | 14 | 11 | 10 | -- | -- |
| 82 | 16 | 18 | 20 | 15 | 13 | 1.2 | -- |
| 83 | 24 | 12 | 13 | 13 | 12 | -- | -- |
| 84 | 16 | 10 | 9 | 9 | 8 | -- | -- |
| 87 | 25 | 21 | 24 | 27 | 28 | 3.0 | 5.2 |
| 88 | 24 | 22 | 23 | 24 | 23 | -- | -- |
| 91 | 28 | 24 | 21 | 12 | 11 | 1.7 | -- |
| 92 | 29 | 28 | 23 | 15 | 14 | 1.5 | 3.7 |
| 93 | 36 | 18 | 15 | 14 | 13 | 0.3 | 3.6 |
| 94 | 11 | 8 | 10 | 10 | 10 | 1.6 | 3.6 |
| 95 | 19 | 13 | 10 | 10 | 12 | 1.0 | 4.0 |
| 96 | 18 | 16 | 12 | 12 | 15 | 1.2 | 5.2 |
| 97 | 19 | 20 | 15 | 13 | 13 | 2.6 | 4.0 |
| 98 | 35 | 17 | 15 | 10 | -- | -- | -- |
| 99 | 20 | 14 | 14 | 12 | 14 | -- | -- |
| 100 | 18 | 11 | 10 | 14 | 14 | 1.2 | 3.7 |
| 101 | 21 | 13 | 11 | 16 | 16 | 3.2 | -- |
| 102 | 15 | 13 | 17 | 10 | 12 | -- | -- |

B29831